

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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| In re Application of: | HABECKER et al. |) | Examiner: | Jie Yang |
| | |) | | |
| Application No.: | 10/795,968 |) | Group Art Unit: | 1793 |
| | |) | | |
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| Docket No.: | 99066CON2 (3600-198-02) |) | | |

For: HIGH CAPACITANCE NIOBIUM POWDERS
AND ELECTROLYTIC CAPACITOR ANODES

APPEAL BRIEF
UNDER 37 C.F.R. § 41

Mail Stop **Appeal Brief – Patents**
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

October 24, 2008

Sir:

(I) Identification

The applicant, application, and the Examiner's identification data associated with this paper are provided in the above-captioned heading.

Appellants hereby file an Appeal Brief under 37 C.F.R. § 41.37, together with the applicable fee under 37 C.F.R. § 41.20(b)(2).

A Notice of Appeal under 37 C.F.R. §41.31 was previously filed with the applicable fee under 41.20(b)(1) on October 6, 2008.

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(3) Real Party in Interest

The real party in interest in this case is *Cabot Corporation*.

(4) Related Appeals and Interferences

Appellants are not aware of any other appeals or interferences that will directly affect, be directly affected by, or have a bearing on the Board's decision in the present appeal.

(5) Status of Claims

Claims 1-35, 59, and 60 are canceled.

Claims 36-58 and 61-65 are rejected.

Claims 36-58 and 61-65 are on appeal.

(6) Status of Amendments

No amendments to the pending claims were submitted after the final rejection.

(7) Summary of Claimed Subject Matter

I. Concise Explanation of the Subject Matter Defined in Independent Claims and Separately Argued Dependent Claims

a) Independent Claim 36

Independent claim 36 is directed to an agglomerated niobium powder (page 4, lines 12-13) characterized by

a capacitance of at least 65,000 CV/g (page 9, lines 10-13; page 17, lines 2-4) and

a DC leakage of less than 5.0 nA/CV (page 10, lines 11-12)

when the agglomerated niobium powder is tested under electrical properties test conditions of sintering at a temperature of 1100° C for 10 minutes (page 9, lines 13-19) and anodizing at a formation voltage of 20Vf at 60° C (page 9, lines 19-20 and page 11, lines 15-30).

b) Dependent Claim 38

Claim 38, which depends from claim 36, further specifies that the capacitance is from about 75,000 to about 175,000 CV/g (page 9, line 12; page 17, lines 7-8).

c) Dependent Claim 39

Claim 39, which depends from claim 36, further specifies that the niobium powder of claim 36, wherein said capacitance is from about 100,000 to about 250,000 CV/g (page 9, lines 10-14 and page 17, line 9-10).

d) Dependent Claim 40

Claim 40, which depends from claim 36, further specifies that the capacitance is from about 125,000 to about 250,000 CV/g (page 9, lines 10-14 and page 17, lines 11-12).

e) Dependent Claim 41

Claim 41, which depends from claim 36, further specifies that the capacitance is from about 100,000 to about 210,000 CV/g (page 9, lines 10-14 and page 17, lines 13-14).

(f) Dependent Claim 44

Claim 44, which depends from claim 36, further specifies that the niobium powder has a BET surface area of at least about $5.5 \text{ m}^2/\text{g}$ (page 4, lines 8-9, 18 and page 17, lines 19-20).

(g) Dependent Claim 57

Claim 57, which depends from claim 36, further specifies that the niobium powder has a particle size of from 5 to 80 microns (page 13, lines 22-23).

(h) Independent Claim 65

Independent claim 65 is directed to an agglomerated niobium powder (page 4, lines 12-13) characterized by

a capacitance of at least 65,000 CV/g (page 9, lines 10-13; page 17, lines 2-4) and

a DC leakage of less than 5.0 nA/CV (page 10, lines 11-12)

when the agglomerated niobium powder is tested under electrical properties test conditions of sintering at a temperature of 1100°C for 10 minutes (page 9, lines 13-19) and anodizing at a formation voltage of 35Vf at 60°C (page 9, lines 19-20; page 11, lines 15-30; and page 12, lines 23-25).

(8) Grounds of Rejection to be Reviewed on Appeal

1) Whether claims 36-43, 50-56, 58, 61, and 65 are unpatentable under 35 U.S.C. § 102(b) as being anticipated by Chang (U.S. Pat. No. 5,448,447), or in the alternative, under 35 U.S.C. § 102(b) as being anticipated by Chang as evidenced by He et al. (U.S. Pat. No. 6,786,951 B2).

2) Whether claims 48, 49, 57, and 62-64 are unpatentable under 35 U.S.C. § 103(a) as being obvious over Chang in view of He et al.

3) Whether claims 36-47, 49-58, and 61-65 are unpatentable under 35 U.S.C. § 103(a) as being obvious over WO 98/37248 (WO '248) in view of Chang, and further in view of He et al.

(9) **Argument**

- 1) **Rejection of Claims 36-43, 50-56, 58, 61, and 65 Under 35 U.S.C. § 102(b) as being unpatentable over Chang ('447) alone, or as evidenced by He et al. ('951 B2).**

Claims 36, 37, 42, 43, 50-56, 58, and 61

In the Final Office Action dated July 9, 2008 (page 3), the Examiner indicated that Chang alone, or, alternatively, as evidenced in view of He et al., was applied to claims 36-43, 50-56, 58, 61, and 65 for the same reason as stated in a previous rejection dated December 28, 2007. The referenced previous Office Action of December 28, 2007 (pages 3-4) set forth the following reasons for this rejection:

'447 is applied to the claims 36-43, 50-56, 58, 61 and 65 for the same reason as state in the previous rejection dated 7/19/2007.^[1]

As to the amendment in claims 36 and 65, regarding "agglomerated" limitation, '447 teaches the powder may be agglomerated by heat treatment and crushed to a certain particulate size (Col. 1, line 61 to Col.2, line 8 and col.4, lines 1-18 of '447). The other amendments in the claims 36 and 65 did not change the claimed scopes. Therefore, '447 anticipates the claimed invention.

Alternatively, '477 [sic] teaches the chemical and physical properties of tantalum and niobium are known by those skilled in the art to be sufficiently similar to permit substitution of either metal (Col. 3, lines 60-68), which is further evidenced by '951. '951 teaches a high surface area tantalum and/or niobium powders via the reduction of the corresponding tantalum and/or niobium oxides for electrolytic capacitors application (Abstract of '951). Under the similar treatment conditions (Examples 1-6 of '951), the Ta and Nb powders have been shown a similar electrical properties (refer to table 6-7 of '951). See MPEP 2112 I&II.

... ..

¹ The Examiner's reasons for relying on Chang alone as the basis of the rejection are not fully set forth in the referenced office action of "7/19/2007", which instead also relies on additional incorporations by reference that thread back through three additional previous offices actions, ultimately to an earlier Office Action of September 26, 2005.

In the Final Office Action dated July 9, 2008 (pages 5-7), the Examiner added the following arguments in further support of this rejection:

... US '477 [sic] clearly teaches the powder may be agglomerated by heat treatment and crushed to a certain particle size (Col. 1, line 61 to Col. 2, line 8 and col. 4, lines 1-28 of US '447 in the previous office action, the quoted lines is typo error as 1-18). US '477 [sic] teaches: "The base materials employed in one embodiment of the present invention were agglomerated by heat treatment while being subject to a vacuum or an inert gas environment. Temperatures in the range of 1200.degree. C. to 1600.degree. C., and preferably in the range of 1400.degree. C. to 1500.degree. C. were used. The heat treatment process may be repeated to achieve a desired degree of agglomeration. Those skilled in the art will recognize the thermal conditions and heating times necessary to achieve a desired level of agglomeration of the selected powder." (col. 4, lines 19-28 of US '447). US '477 [sic] (Chang) discloses that chemical and physical properties of tantalum and niobium are known by those skilled in the art to be sufficiently similar to permit substitution of either metal (Col. 3, lines 60-68 of US '477 [sic]).

... As pointed out in the previous office action marked 12/28/2007, US '951 (He et al.) is used as a factual reference (i.e., evidence ref.), it does not need antedate the filing date. MPEP 2124. US '477 [sic] (Chang) teaches: "According to one embodiment of the present invention, capacitor powder for low leakage capacitors is produced from base materials which contain at least one metal powder selected from Group V-B of the Periodic Table. For simplicity purposes, reference shall be made to tantalum metal hereafter even though the chemical and physical properties of tantalum and niobium are known by those skilled in the art to be sufficiently similar to permit substitution of either metal." (Col. 3, lines 60-68 of US '951), which clearly support the Examiner's position that US '477 teaches the niobium powder. As pointed out in the previous office action marked 12/28/2007, US '951 teaches a high surface area tantalum and/or niobium powders via the reduction of the corresponding tantalum and/or niobium oxides for electrolytic capacitors application (Abstract of '951). Under the similar treatment conditions (Examples 1-6 of '951), the Ta and Nb powders have shown the similar electrical properties (refer to table 6-7 of '951). See MPEP 2112 I&II. The Applicants' argument does not show unexpected different results between Ta and Nb powders. Therefore, US '447, or in the alternative, US '477 in view of US '951 is applied to the claims 36-43, 50-56, 58, 61 and 65 are proper.

Claim 36 of the present application recites an agglomerated niobium powder having a capacitance of at least 65,000 CV/g and a DC leakage of less than 5.0 nA/CV when the agglomerated niobium powder is tested under electrical properties tests conditions of sintering at a temperature of 1100° C for 10 minutes and anodizing at a formation voltage of 20Vf at 60° C. Claim 36 thus recites electrical characteristics of a sintered agglomerated niobium powder, and also test conditions for determining those recited electrical characteristics of the sintered agglomerated powder. These aspects of the invention are clearly described in the present application, *e.g.*, at pg. 8, line 20 to pg. 9, line 3. As also described in the present application, in the past, others in the industry had failed to develop niobium powders having electrical characteristics that might be a suitable replacement for tantalum (*e.g.*, see pg. 1, line 15 to pg. 2, line 2). In particular, others had concluded niobium was not capable of replacing tantalum in the electrolytic capacitor market in particular. Where others have failed or discouraged efforts, the present appellants have succeeded in discovering agglomerated niobium powders having high capacitance capability and low DC leakage, such as recited in claim 36 on appeal. Although not the subject matter currently on appeal, the present appellants also have developed unique techniques, which are described in the present application for obtaining the presently claimed high capacitance, low DC leakage niobium powders.² This bears mentioning as the Examiner's anticipation rejections are premised on theories of inherency, as will be addressed in greater detail below.

² Appellants' parent appln., U.S. Patent Application No. 09/310,322, which issued as U.S. Pat. No. 6,375,704 B1, includes, *inter alia*, claim 24 directed to a method for making a flaked niobium powder comprising milling niobium chips to form flaked niobium powder and then subjecting the flaked niobium powder to deoxidation and then continuing milling of the flaked niobium powder.

Chang Does Not Identically Teach Agglomerated Niobium Powders as Required By Claim 36

The proper standard for anticipation is explained in M.P.E.P. §2131, which states, in relevant part:

"A claim is anticipated only if each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference." *Verdegaal Bros. v. Union Oil Co. of California*, 814 F.2d 628, 631, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987) ...

The elements must be arranged as required by the claim ... *In re Bond*, 910 F.2d 831, 15 USPQ2d 1566 (Fed. Cir. 1990).

Thus, the MPEP thus instructs that a proper anticipation rejection requires that the single prior art reference teach *the elements arranged as required by the claim*. This means that the asserted anticipating reference must clearly and unequivocally disclose the subject matter of the rejected claims or direct those skilled in the art to the subject matter without *any* need for picking, choosing, and combining various disclosures not directly related to each other by the teachings of the cited reference. *In re Arkley*, 455 F.2d 586, 587 (CCPA 1972).

With respect to the Examiner's argument that Chang shows agglomerated type tantalum or niobium powders, the Examiner's evidence for this assertion in Chang is based upon col. 1, line 61 to col. 2, line 8, and also col. 4, lines 1-28 of Chang. However, these teachings of Chang do not support the Examiner's position on anticipation based on Chang. In particular, col. 1, line 61 to col. 2, line 8, is the background section of Chang and specifically refers to a description of U.S. Patent No. 4,544,403 and, therefore, it is not the invention of Chang. Put another way, the very description relied upon by the Examiner for agglomerated powders is a different powder than the material described in Chang and for which the Examiner is basing this rejection upon. In essence, the Examiner is mixing and matching different inventions, whereas Chang clearly does not describe

or suggest that the embodiments of U.S. Patent No. 4,544,403 can be incorporated into Chang's invention. Moreover, in this section of Chang (*i.e.*, col. 1, line 61 to col. 2, line 8), U.S. Patent No. 4,544,403 strictly relates to tantalum-based materials and not at all to niobium. Therefore, this section would clearly teach that one can agglomerate certain types of tantalum-based materials, but not niobium and, therefore, does not support the Examiner's anticipation rejection.

The Examiner's alternate reliance on col. 4, lines 1-28 of Chang is based on an incomplete reading of the teachings of the Chang reference. This particular section of Chang, in part, mentions the agglomeration of powders (especially, col. 4, lines 19-28), as a possible treatment of base materials formed by chemical reduction of tantalum-bearing salts or the crushing of tantalum-bearing ingots. The Examiner, however, disregarded the teachings of Chang at col. 4, lines 34-36, which immediately follows the discussion of possible agglomerated forms of tantalum powder. Chang therein states it is within the scope of the invention to prepare electrodes from powders "... which have not been subject to agglomeration heat treatment." Thus, the general teachings of Chang presented at col. 4, lines 1-35, when properly read as a whole, characterize agglomeration of the powders as being optional and not necessary. The examples described at columns 7-11 of Chang also do not require that the base nodular tantalum powder materials be agglomerated as a necessary feature. Further, the examples described in Chang are not directed to niobium powders. They are tantalum powders.

The Examiner also has cited and relied on col. 3, lines 60-68 of Chang as teaching or suggesting that the subsequent references made exclusively to tantalum metal within the Chang reference, such as col. 4, lines 1-28, were for simplicity purposes only as the chemical and physical properties of tantalum and niobium are known by those skilled in the art to be sufficiently similar to permit substitution of either metal. Col. 3, lines 60-68 of Chang et al. is reproduced below:

According to one embodiment of the present invention, capacitor powder for low leakage capacitors is produced from base materials which contain at least one metal powder selected from Group V-B of the Periodic Table. For simplicity purposes, reference shall be made to tantalum metal hereafter even though the chemical and physical properties of tantalum and niobium are known by those skilled in the art to be sufficiently similar to permit substitution of either metal.

However, even if for sake of argument only, niobium is “substituted” for recitations of tantalum in teachings subsequent to col. 3, lines 60-68 of Chang, those modified powders still would not necessarily be agglomerated, as explained above. Furthermore, there is no specific single teaching or example in Chang of agglomerated *and* niobium type powders in combination. To reach that hypothetical combination would require choosing niobium instead tantalum and also choosing agglomerated form instead of non-agglomerated form powders. It is noted that the Chang passage does not state that the tantalum and niobium have “similar” electrical properties.

To support a rejection under 35 U.S.C. §102(b), Chang must teach literally or inherently each and every limitation set forth in the claims on appeal. In view of the above, Chang *per se* fails to disclose agglomerated niobium powders as arranged as required in claim 36. Therefore, for at least this reason, Chang *per se* does not anticipate the claim, or any claims dependent thereon.

Chang Is Missing The Electrical Properties For Niobium Powders Required By Claim 36

Claim 36 of the present application also recites that the agglomerated niobium powder is characterized by electrical properties of a capacitance of at least 65,000 CV/g and a DC leakage of less than 5.0 nA/CV, when the agglomerated niobium powder is tested under electrical properties test conditions of sintering at a temperature of 1100° C for 10 minutes and anodizing at a formation voltage of 20Vf at 60° C.

As indicated, Chang does not recite any examples whatsoever of niobium. Further, Chang

does not provide chemical characteristics, and does not provide any physical characteristics, and most importantly, does not provide any electrical characteristics of any niobium powder. At col. 3, lines 60-68 thereof, Chang does not state that the niobium electrical characteristics are the same as the tantalum electrical characteristics and, further, Chang does not state that tantalum electrical characteristics can be substituted as equivalent to niobium electrical characteristics.

Chang describes leakage and specific capacitance electrical properties *only* for tantalum powders (see, Figs. 1-4; col. 3, lines 1-59). As indicated, the Examiner has relied on the teachings at col. 3, lines 60-68 in Chang as teaching that the chemical and physical properties of tantalum and niobium are known by those skilled in art to be sufficiently similar to permit substitution of either metal. However, Chang does not teach or suggest that the electrical properties of niobium and tantalum are sufficiently similar to permit substitution of either metal on that basis. That is to say, Chang does not teach, suggest or imply that any leakage and capacitance electrical characteristics of tantalum materials that are actually described in the Chang reference would apply to niobium materials substituted for them, even if produced by identical or substantially identical processes as those described in the Chang reference for tantalum.

Further, col. 3, lines 1-10 of Chang states that sintering is done between 1400° C to 1800° C and the anodization voltage is 100 volts or greater in making electrode capacitors according to the teachings of the reference. Also, the highest reported capacitance in Chang is about 25,000 CV/g (*e.g.*, see Fig. 2; Example 10, Table 2). Chang fails to teach a sinter temperature lower than 1400° C, an anodization voltage of lower than 100 volts, or a capacitance of greater than 25,000 CV/g, such as recited in claim 36 on appeal. It is clear that Chang does not teach or suggest the electrical testing conditions prescribed in claim 36. One skilled in the art would understand this to mean that the powder of Chang is not sinterable at below 1400° C, that the powder cannot be anodized at a

voltage of less than 100 volts, or that the material is capable of having a capacitance above 25,000 CV/g. Certainly, Chang would provide no motivation to even try these parameters. A reference is viewed by one skilled in the art and what teachings or suggestions are presented as a whole in the reference. Clearly, Chang, when read in its entirety, does not provide any such suggestions with regard to any metal powder that is capable of being subjected to a lower sinter temperature or lower anodization formation voltage, or being capable of achieving a higher capacitance other than identified above. Further, there is no teaching or suggestion in Chang of the DC leakage of less than 5.0 nA/CV as recited in present claim 36. Contrary to the teachings and suggestions of Chang, the present niobium powder is capable of being sintered at a temperature of 1100° C and is capable of being anodized using a formation voltage of, for instance, 20 Vf and has a capacitance of at least 65,000 CV/g and further has a DC leakage of less than 5.0 nA/CV. These differences alone clearly show that Chang does not teach or suggest the claimed invention.

In addition, the Declaration under 37 C.F.R. §1.132 of Heather Enman filed with the appellants' response on February 24, 2006 ("the 2006 Enman Declaration"), a copy of which is included in the Evidence Appendix section of this brief, provides further evidence showing that the Examiner's substitution argument would not have merit as applied to electrical properties of tantalum and niobium. The 2006 Enman Declaration evidence shows that even if one was capable of using the particular chemical and physical characteristics of tantalum in a niobium powder, one would not achieve the particular niobium powder recited in the claims as shown by the significant difference in electrical properties. In this 2006 Enman Declaration, niobium samples having a BET of 0.58 to 0.71 m²/g when formed at a formation voltage of 35 volts, at a formation temperature of 60° C, and at sintered temperatures of 1,400 ° C or 1,300° C, for a sinter time of 10 minutes. While these conditions are not identical to the test standards set forth in present claim 36, they are

sufficiently close enough from a technical standpoint to show that a niobium powder having a BET in the range of about 0.25 and about 0.55 m²/g disclosed by Chang (*e.g.*, see col. 5, lines 54-56; claim 31), simply would not have the capacitance recited in claim 36 and the claims dependent thereon. In addition, a graph was included in the 2006 Enman Declaration to extrapolate the capacitance to a sintered temperature 1,100 °C based on a linear relationship. This particular graph was done for a BET of 0.58 m²/g, and the capacitance would be about 40,000 CV/g at 1,100° C. This is significantly below the lower limit of 65,000 CV/g stated in claim 36. The Examiner has questioned how this extrapolation is acceptable. First, in the 2006 Enman Declaration, a person skilled in the art having many years of experience in tantalum and niobium research and development has stated that this extrapolation practice is an acceptable practice in this field of technology. The Examiner has provided no other evidence to contradict such a statement. Furthermore, a brochure from a competitor of the applicant, Starck, has been made of record, and a copy of which is included in the Evidence Appendix of this brief as the “the Starck Brochure”, where it is shown that the relationship for capacitance and sintering temperature is generally accepted as a linear relationship (for instance, see the graph for PL-8000). This is further evidence to show that the extrapolations made in the 2006 (and 2007) Declarations by Heather Enman of record are acceptable and are common practice in the industry. In addition, even if a fair degree of non-linearity were to exist in the CV/g--sintered temperature relationship, as set forth in the 2006 Enman Declaration, the capacitance for the 0.58 m²/g BET sample would still be below 65,000 CV/g in the technical opinion of the declarant based her work and experience in the niobium powder area. This evidence clearly shows that the Examiner's assumption that niobium powder can be substituted for tantalum powder would not provide the type of niobium powder set forth in the claims of the present application with respect to electrical properties.

Also, another Declaration under 37 C.F.R. §1.132 by Heather Enman filed with the appellants' response on April 30, 2007 ("the 2007 Enman Declaration"), a copy of which is also included in the Evidence Appendix section of this brief, shows through extrapolation and additional data that even if the Examiner's proposed substitution of niobium for tantalum powder was proper, for sake of argument, the niobium powder having the BET surface area of Chang and tested at electrical formation conditions of 20 volts and a sinter temperature of 1100° C would still lead to a niobium powder having a capacitance of less than 65,000 CV/g. Thus, the use of a formation voltage of 35 volts (such as used in the tests of the 2006 Enman Declaration) versus 20 volts (as used in the tests of the 2007 Enman Declaration) did not and would not significantly affect the capacitance to the extent that a powder of 0.58 m²/g would achieve a capacitance of at least 65,000 CV/g.

In addition, with further reference to the 2007 Enman Declaration, it is further pointed out that if the Examiner's substitution argument is correct, for sake of argument only, then one skilled in the art should be able to take the tantalum powder of Chang, and using the testing conditions set forth in claim 36, obtain the electrical properties set forth in claim 36. In other words, according to the Examiner's substitution argument, if one takes the tantalum powder used in Chang and tests for electrical properties by sintering at a temperature of 1100° C for 10 minutes and anodized using a formation voltage of 20 Vf at 60° C, one would achieve a capacitance of at least 65,000 CV/g and a DC leakage of less than 5.0 nA/CV. However, as shown in the 2007 Enman Declaration, tantalum powder, having a BET of less than 0.6 m²/g, would be essentially incapable of forming any capacitance near 65,000 CV/g, since it is such a low sintering temperature, the tantalum powder of Chang would be incapable of forming an anode to obtain capacitance and the DC leakage would be well above 5 nA/CV. This evidence further shows the inability to make the substitution alleged and

proposed by the Examiner in the final rejection.

In addition, the Declaration under 37 C.F.R. §1.132 of Jonathon L. Kimmel filed with the appellants' response on April 30, 2007 ("the Kimmel Declaration"), a copy of which is also included in the Evidence Appendix section of this brief, further discusses and provides evidence on why niobium is not a substitute for tantalum with respect to electrical properties and other features. Furthermore, the appellants note that if the substitution of niobium for tantalum was made, as proposed by the Examiner, niobium powder would have been used commercially as a substitute for tantalum anodes some time ago. However, the first commercial niobium anode made with niobium powder was only recently introduced commercially, well after the §120 filing date of the present application.

Therefore, this evidence of record rebuts and shows that the Examiner's position is incorrect that niobium powder is a substitute for, or interchangeable with, tantalum powder, especially with respect to electrical properties, and also rebuts the Examiner's position that Chang would permit one to make such a substitution and achieve the particular niobium powder set forth in the claims on appeal.

He et al. Is Not Prior Art To The Present Application

The Examiner has alternatively relied on Chang "as evidenced" by He et al. in finding that claim 36 is anticipated by Chang.

With respect to the Examiner's reliance on He et al., it initially is pointed out that He et al. is not prior art to the present invention. The Examiner has not disputed this fact. He et al. was granted September 7, 2004, and has a publication date (with respect to its application) of June 19, 2003, and has a U.S. filing date of September 30, 2002. However, the present invention, through

several continuation applications, claims the benefit of U.S. Patent Application No. 09/310,322 filed May 12, 1999. Therefore, He et al. is not prior art to the present invention. Citing M.P.E.P. § 2124, the Examiner has argued that He et al. is only used as a factual reference or evidence reference, and thus does not need to antedate the effective filing date of the present application. The appellants respectfully submit that He et al. does not cure the deficiencies of the Chang reference, even if applied in an appropriate manner as a “factual or evidence reference” only, for the following reasons.

He et al. Fails to Show That Missing Electrical Properties in Chang Are Necessarily Present As Inherent Features of Chang's Powders

The Examiner is correct that M.P.E.P. § 2124 indicates that, in certain circumstances, references cited to show “a universal fact” need not be available as prior art before applicant’s filing date. Further, M.P.E.P. 2131.03 (III) explains, in relevant part, that an extra reference or evidence can be used to show an inherent characteristic of the thing taught by the primary reference under the following conditions:

‘To serve as an anticipation when the reference is silent about the asserted inherent characteristic, such gap in the reference may be filled with recourse to extrinsic evidence. Such evidence must make clear that the missing descriptive matter is necessarily present in the thing described in the reference, and that it would be so recognized by persons of ordinary skill.’ *Continental Can Co. USA v. Monsanto Co.*, 948 F.2d 1264, 1268, 20 USPQ2d 1746, 1749 (Fed. Cir. 1991) ...

Under these guidelines, He et al. fails to show and make clear that the electrical properties recited in claim 36 on appeal are *necessarily present* in any powders *described in Chang*.

An inherent disclosure in a prior art reference requires that the characteristic is a “necessary

feature or result of a prior-art embodiment" that is sufficiently described and enabled. If one looks at Chang, the Examiner has not explained what embodiment of Chang has this inherent feature (i.e., the electrical properties of claim 36). As indicated, all of the examples of Chang relate to tantalum powder, and Chang does not provide any particular example of niobium powders. Therefore, there is no part of Chang which would permit the Examiner to take the position that the parameters set forth in the pending claims are a necessary feature or result of a prior-art embodiment in Chang. In particular, the Examiner has failed to show that it is a universal fact that if niobium is substituted for tantalum in Chang, that the niobium powders will have the same electrical properties as the tantalum powders.

Further, the Examiner never shows, and cannot show, that Examples 1-6 of He et al. make powders by the same or similar treatments conditions taught by Chang. Chang teaches making tantalum base materials by chemical reduction of potassium tantalum fluoride salt by metallic sodium or hydrating a tantalum ingot before crushing it into base material (col. 4, lines 1-18). Chang then deoxidizes the base material using magnesium, such as described in the examples. He et al. specifically *teaches away* from producing tantalum powder by reducing potassium fluorotantalate with sodium (see col. 1, lines 46-65). Instead, He et al. reduces tantalum oxide or niobium oxide as starting material in the presence of metal halide selected from halides of Mg, Ca, Sr, Ba and Ce and alkali metal (abstract; Examples 1-6). Clearly, Chang and He et al. teach forming powders by materially different process strategies and steps. Thus, there is no presumptive correlation that processes of He et al. and Chang form substantially the same product where their respective processes materially differ from one another. *Ashland Oil, Inc. v. Delta Resins & Refractories, Inc.*, 776 F.2d. 281, 227 USPQ 657 (Fed. Cir. 1985).

Further, though not prior art, the appellants also point out that He et al. does not show

tantalum and niobium powders having similar electrical properties since the Examiner has not taken into account the particular properties of the powders being used to form the anodes which have been tested in Tables 6 and 7 of He et al. It is respectfully noted that Table 6 shows electrical properties of tantalum powders and Table 7 shows electrical properties of niobium powders; however, when one goes back into the He et al. patent to see the particular powder properties, one notes that in Examples 1, 2, and 3, the tantalum powders have a significantly different Scott bulk density, a significantly different particle size (FSS), and, most importantly, a very different BET surface area. For instance, the BET surface area of the tantalum powders is from 2.1 m²/g to 3.8 m²/g, whereas the BET surface area of the niobium powders that were tested had a BET surface area of 4.9 m²/g to 5.6 m²/g in Examples 6 and 5, and a BET surface area of 26.72 m²/g in Example 4. In addition, the Green density and sintered density were dramatically different from the test conditions used for the tantalum powder as shown in Tables 6 and 7. Thus, the Examiner's assertion that "similar treatment conditions" were used is technically incorrect as shown above and no conclusions can be drawn from the He et al. reference. Also, it is important to point out that Chang shows a BET surface area of no more than 0.6 m²/g (*see* col. 5, line 55), whereas He et al. used a BET surface area of 2 m²/g and above -- very different powders exist in He et al., and such data is irrelevant to the low surface area powder of Chang.

Thus, the Examiner's reliance on Examples 1-6 of He et al. for extrinsic evidence on the possible electrical properties of the powders of Chang is improper because of the material divergence in their respective methods of manufacture, and non-uniformity of Ta and Nb data within the examples of He et al., disallows any reasonable technical conclusion that the electrical properties of one product is "necessarily present" in the other. The Examiner's reliance on MPEP 2112, §I and §II, as supporting the "inherency rejection" based on He et al. is thus misplaced. As

instructed in M.P.E.P. § 2112 (IV), which is not acknowledged by the Examiner, the examiner must provide a rationale or evidence tending to show inherency in compliance with the following legal precedents:

The fact that a certain result or characteristic may occur or be present in the prior art is not sufficient to establish the inherency of that result or characteristic. *In re Rijckaert*, 9 F.3d 1531, 1534, 28 USPQ2d 1955, 1957 (Fed. Cir. 1993) ...; *In re Oelrich*, 666 F.2d 578, 581-82, 212 USPQ 323, 326 (CCPA 1981). "To establish inherency, the extrinsic evidence 'must make clear that the missing descriptive matter is necessarily present in the thing described in the reference, and that it would be so recognized by persons of ordinary skill. Inherency, however, may not be established by probabilities or possibilities. The mere fact that a certain thing may result from a given set of circumstances is not sufficient.' " *In re Robertson*, 169 F.3d 743, 745, 49 USPQ2d 1949, 1950-51 (Fed. Cir. 1999) (citations omitted) ...

Whatever the non-prior art reference to He et al. may teach about the electrical properties of tantalum and niobium powders *as made* according to the teachings of that reference, they cannot be assumed to apply to the powders of Chang, which are made by a materially different process. He et al. thus fails to teach a "universal fact" that applies to the electrical properties of the powders of Chang as a matter of certainty, and not mere probabilities or possibilities. Chang as evidenced by He et al. thus fails to meet the above-indicated criteria for anticipation by inherency.

In any event, as shown with evidence in the Enman and Kimmel Declarations discussed above, the electrical properties of the tantalum powders exemplified in Chang itself do not fall within the capacitance and DC leakage recitations of present claim 36 on appeal. He et al. does not rebut that evidence and cannot be used to modify the primary reference as an "evidence reference."

In view of at least these reasons, Chang alone or evidenced by He et al. does not establish

the *prima facie* anticipation of claim 36, nor any of its dependent claims, on appeal.

For these reasons, this rejection should be reversed.

Claims 38-41

Claims 38-41 on appeal are each dependent on claim 36. Claim 38 further specifies that the capacitance is from about 75,000 to about 175,000 CV/g. Claim 39 further specifies that the niobium powder of claim 36, wherein said capacitance is from about 100,000 to about 250,000 CV/g. Claim 40 further specifies that the capacitance is from about 125,000 to about 250,000 CV/g. Claim 41 further specifies that the capacitance is from about 100,000 to about 210,000 CV/g.

The reasons for reversal as discussed above with respect to the rejection of parent claim 36 apply equally to claims 38-41.

In addition, as indicated, the 2007 Enman Declaration shows through extrapolation and additional data that even if the Examiner's proposed substitution of niobium for tantalum powder was proper, for sake of argument, the niobium powder having the BET surface area of Chang and tested at electrical formation conditions of 20 volts and a sinter temperature of 1100° C would still lead to a niobium powder having a capacitance of less than 65,000 CV/g. The capacitance ranges recited in each of claims 38-41 is even further significantly separated from and different in kind from the capacitance values of Chang powders.

He et al. does not rebut that evidence and cannot be used to modify the primary reference as an "evidence reference."

In view of the above reasons, this rejection of claims 38-41 also should be reversed.

Claim 65

Independent claim 65 has the same language as claim 36, but recites a formation voltage of 35Vf. The reasons for reversal as discussed above with respect to the rejection of claim 36 apply equally here. Additional reasons for reversal are as follows.

As indicated above, the 2006 Enman Declaration shows through extrapolation and additional data that even if the Examiner's proposed substitution of niobium for tantalum powder was proper, for sake of argument, the niobium powder having the BET surface area of Chang and tested at electrical formation conditions of 35 volts and a sinter temperature of 1100° C would still lead to a niobium powder having a capacitance of significantly less than 65,000 CV/g stated in claim 65.

As indicated above, He et al. does not rebut that evidence and cannot be used to modify the primary reference as an "evidence reference."

In view of at least these reasons, Chang alone or evidenced by He et al. does not establish the *prima facie* anticipation of claim 65, nor any of its dependent claims, on appeal.

In view of the above reasons, this rejection of claim 65 also should be reversed.

2) Rejection of Claims 48, 49, 57, and 62-64 Under 35 U.S.C. § 103(a) as being unpatentable over Chang ('447) in view of He et al. ('951 B2).

Claims 48, 49, and 62-64

In the Final Office Action dated July 9, 2008 (pages 3-4), the Examiner indicated that the descriptions of Chang, alone or as evidenced in view of He et al., were applied to claims 48, 49, 57, and 62-64 for the same reason as stated in the previous rejection dated July 19, 2007, which in turn references an earlier Office Action of October 30, 2006. In the earlier Office Action of October 30, 2006 (page 6), the Examiner had asserted that Chang teaches the oxygen content

recited in claims 48 and 49. The Examiner also indicated that He et al. was applied to claims 48, 49, 57 and 62-64 as a new evidence for the same reasons as discussed in the a §102/§103 rejections for independent claim 36.

For the reasons set forth above, this rejection should also be reversed. It is noted that these claims are dependent ultimately on claim 36 and, therefore, recite a niobium powder having certain electrical characteristics in addition to the various limitations set forth in the rejected dependent claims. As stated, Chang does not teach or suggest a niobium powder having the characteristics set forth in the claims, and the Examiner's proposed substitution argument regarding tantalum and niobium is incorrect and does not achieve niobium powders as claimed in claim 36.

In addition, Chang, at col. 6, lines 13-18, refers to a synergistic effect of reacting quantities of oxygen and nitrogen with base material which results in a more stable dielectric layer of tantalum pentoxide. This particular paragraph at col. 6 further states the effect of various impurities with tantalum or tantalum oxide. Thus, Chang specifically teaches a synergistic effect with oxygen, nitrogen, and tantalum, but makes no mention whatsoever of niobium. Again, this is further evidence that Chang had an understanding to some extent of tantalum, but had no understanding and no data to gain an understanding of niobium and its effects.

Furthermore, claim 36, from which claims 48, 49, and 62-64 depend ultimately, recites a DC leakage of less than 5.0 nA/CV. Chang does not disclose such a DC leakage value for the powders disclosed therein. The appellants note that in Fig. 1 of Chang, this figure would suggest to one skilled in the art that lower formation voltage (of even 100 volts) leads to higher leakage. Fig. 1 of Chang plots a percentage reduction in leakage versus a formation voltage. In Fig. 1, the lowest formation voltage shown is 100 volts and nothing less. Second, it is clear from Fig. 1 that as the formation voltage is decreased from 400 volts to 100 volts, the percent reduction in leakage

decreases as well. In other words, the amount of leakage is *increasing* as the formation voltage is dropping. Thus, contrary to the positions taken by the Examiner in rejecting these claims, Chang actually teaches that leakage will increase dramatically as formation voltage drops and, therefore, the powders of Chang actually *teach away* from being capable of a lower formation voltage and yet obtaining satisfactory DC leakage and capacitance.

As instructed in M.P.E.P. § 2141.02:

A prior art reference must be considered in its entirety, i.e., as a whole, including portions that would lead away from the claimed invention. *W.L. Gore & Associates, Inc. v. Garlock, Inc.*, 721 F.2d 1540, 220 USPQ 303 (Fed. Cir. 1983), *cert. denied*, 469 U.S. 851 (1984)...

The appellants submit that the Examiner, in making this obviousness rejection of claims 48, 49, and 62-64 based on Chang, has not considered the entire teachings of the reference, including those portions that would lead away from the claimed invention. In addition, the present invention is not a predictable solution in view of the entire teachings of Chang.

Further, as stated above, He et al. is not prior art to the claimed invention and, therefore, He et al. cannot be relied upon for any teachings or suggestions of possible modifications to the powders of Chang.

Accordingly, this rejection should be reversed.

Claim 57

Claim 57, which depends from claim 36, further specifies that the niobium powder has a particle size of from 5 to 80 microns.

The reasons for reversal as discussed above with respect to the rejection of parent claim 36 apply equally to claim 57. Additional reasons for reversal are as follows.

Chang only provides limited information on powder sizes. Chang refers to agglomerates that can be reduced or crushed down to a powder having a screen size of -40 mesh (e.g., see col. 7, lines 46-50; col. 12, lines 32-33; col. 14, lines 9-10). As well known, a mesh size of 40 mesh refers to a sieve tray having 420 μm sieve openings. Therefore, Chang appears to generally indicate that the agglomerates can be reduced to powder having a particle size of less than 420 μm (i.e., -40 mesh). However, Chang provides no additional details on the *distribution* of the particle sizes of the powder in the broad range of less than 420 μm . There is no certainty that Chang necessarily provides for any powders having a particle size of from 5 to 80 microns. That conclusion would require speculation. Therefore, the recitation in claim 57 of niobium powder having a particle size of from 5 to 80 microns is not obvious over Chang.

Further, as stated above, He et al. is not prior art to the claimed invention and, therefore, He et al. cannot be relied upon for any teachings or suggestions of possible modifications to the powders of Chang.

Accordingly, this rejection should be reversed.

- 3) Rejection of Claims 36-47, 49-58, and 61-65 Under 35 U.S.C. § 103(a) as being unpatentable over WO 98/37248 in view of Chang ('447), and further in view of He et al. ('951 B2).**

Claims 36-43, 45-47, 49-56, 58, and 61-64

In the Final Office Action dated July 9, 2008 (page 4), the Examiner indicated that the descriptions of WO 98/37248 ("WO '248") in view of Chang, and further in view of He et al., were applied to claims 36-47, 49-58, and 61-65 for the same reason as stated in the previous rejection dated July 19, 2007, which in turn references an earlier Office Action of October 30, 2006 (apparently, the discussions of that rejection at pages 6-9 thereof would apply here). The

Examiner essentially relies on Chang for the reasons set forth above. The Examiner asserts that WO '248 shows tantalum powder having a particular capacitance. The Examiner does acknowledge that WO '248 does not teach or suggest that the powder is niobium, but the Examiner asserts that it would be obvious to use niobium in the sintered anodized powder electrodes disclosed by WO '248. As to He et al., the Final Office Action of July 9, 2008, at page 7, appears to suggest that He et al. is being applied to this rejection as new evidence for the same reason as discussed in the a §102/§103 rejections for independent claim 36.

The appellants' comments regarding Chang above apply equally here to this rejection. WO '248 strictly relates to tantalum and makes absolutely no suggestion regarding niobium powder. All of the data, all of the examples, all of the test methods, and the electrical properties only relate to tantalum powder and not niobium powder. The Examiner has essentially taken the position that it would be obvious to take these electrical properties for a completely different powder and routinely obtain these properties for niobium powder in Chang, even though Chang does not specifically describe any niobium examples, and Chang does not describe any niobium characteristics, and only makes a passing reference to niobium powder having similar chemical and physical properties. As indicated, Chang does not state that electrical characteristics are interchangeable with the two powders, nor does WO '248. Therefore, the Examiner's proposed substitution of electrical properties is not supported in any of the cited art relied upon by the Examiner.

Moreover, Chang specifically describes powders below $0.6 \text{ m}^2/\text{g}$ for purposes of all of the information set forth in Chang. Even if the Examiner's proposed substitution argument is acceptable, which the appellants respectfully disagree with, Chang presents this information with respect to low surface area tantalum powder, namely below $0.6 \text{ m}^2/\text{g}$. It is respectfully noted that the BET surface area of the powder set forth in WO '248 ranges from 1.5 to $10 \text{ m}^2/\text{g}$ and most

preferably 3 to 6 m²/g (p. 5, lines 15-16), a difference in size of almost 300% compared to Chang.³ Therefore, the powder described in Chang, even with respect to the tantalum in WO '248, at least with respect to surface area, is completely different.

Further, as indicated, one skilled in niobium powder would not look to the alleged high capacitance tantalum powder set forth in WO '248 to develop niobium powders. As shown in the evidence provided by the 2006 Enman Declaration, the 2007 Enman Declaration, the Kimmel Declaration and Starck Brochure, the types of powder set forth in Chang simply would not achieve the capacitance set forth in the present claims.

As also stated above, He et al. is not prior art to the claimed invention and for this reason alone, this rejection should be reversed. Furthermore, the deficiencies and problems with the Examiner's reliance on He et al., as set forth above, apply equally here.

For these reasons, this rejection should be reversed.

Claim 44

Claim 44, which depends from claim 36, further recites that the niobium powder has a BET surface area of at least about 5.5 m²/g.

The reasons for reversal as discussed above with respect to the rejection of parent claim 36 apply equally to claim 44. Additional reasons for reversal are as follows.

"... [A] patent composed of several elements is not proved obvious merely by demonstrating that each of its elements was, independently, known in the prior art." *KSR Int'l v. Teleflex Inc.*, 127 S. Ct. 1727, 1741 (2007). In rejecting claim 44 on appeal, the Examiner is understood to rely on the high BET specific surfaces of 1.5 to 10 m²/g of the tantalum powder set

³ An English-language equivalent of WO '248 is U.S. Pat. No. 6,193,779 B1 (see col. 3, lines 41-43).

forth in WO '248, and relies separately on Chang for alleged teachings on substitutions of niobium for tantalum.

As indicated above, Chang specifically describes powders that have a low BET surface area below $0.6 \text{ m}^2/\text{g}$ for purposes of all of the information set forth in Chang (e.g., see col., 5, lines 54-58; Tables 1-3 at cols. 8, 10 and 11: Examples 1-14). As also indicated above, the BET specific surfaces of 1.5 to $10 \text{ m}^2/\text{g}$ of the tantalum powder set forth in WO '248 differs significantly compared to BET surface areas of tantalum powders disclosed by Chang.

The Examiner can not properly arbitrarily pick and choose those parts of Chang that may support this rejection, such as alleged niobium and tantalum substitutions, while ignoring other parts that differ and teach away from the combination, such as Chang's teachings on the BET surface areas that should be provided in the product powders that diverge from those of WO '248. "It is impermissible within the framework of section 103 to pick and choose from any one reference only so much of it as will support a given position, to the exclusion of other parts necessary to the full appreciation of what such reference fairly suggests to one of ordinary skill in the art." *In re Wesslau*, 353 F.2d 238, 241, 147 USPQ 391, 393 (CCPA 1965).

As indicated above, He et al. is not prior art to the claimed invention and for this reason alone it thus can not cure the above-indicated deficiencies of WO '248 and Chang with regard to claim 44. Furthermore, the deficiencies and problems with the Examiner's reliance on He et al., as set forth above, apply equally here.

For these reasons, this rejection should be reversed.

Claim 57

As indicated above, claim 57, which depends from claim 36, further specifies that the niobium powder has a particle size of from 5 to 80 microns.

The reasons for reversal as discussed above with respect to the rejection of parent claim 36 based on WO '248, Chang and He et al. apply equally to claim 57. Additional reasons for reversal are as follows.

WO '248 does not teach a particle size for niobium powders. WO '248 only teaches particle sizes for tantalum powders, such as average secondary particles sizes of 10 to 50 μm or more obtained by crushing and sieving (page 2, line 9-19 and page 4, lines 24-29). WO '248 does not suggest that such particle sizes would be equally applicable to niobium powders.

As indicated above, with respect to particle size information, Chang only refers to agglomerates that can be reduced or crushed down to a powder having a screen size of -40 mesh (i.e., $< 420 \mu\text{m}$). Chang provides no additional details on the distribution of the particle sizes of the powder in the broad range of less than 420 μm . There is no certainty that Chang necessarily provides for any powders having a particle size of from 5 to 80 microns. Therefore, the recitation in claim 57 of niobium powder having a particle size of from 5 to 80 microns is not obvious over WO '248 in view of Chang.

As indicated above, He et al. is not prior art to the claimed invention and for this reason alone it thus can not cure the above-indicated deficiencies of WO '248 and Chang with regard to claim 57. Furthermore, the deficiencies and problems with the Examiner's reliance on He et al., as set forth above, apply equally here.

For these reasons, this rejection should be reversed.

Claim 65

As indicated above, independent claim 65 has the same language as claim 36, but recites a formation voltage of 35Vf. The reasons for reversal as discussed above with respect to the rejection of claim 36 apply equally here. Additional reasons for reversal are as follows.

As indicated above, the 2006 Enman Declaration shows through extrapolation and additional data that even if the Examiner's proposed substitution of niobium for tantalum powder was proper, for sake of argument, niobium powder having the BET surface area of Chang and tested at electrical formation conditions of 35 volts and a sinter temperature of 1100° C would still lead to a niobium powder having a capacitance of significantly less than 65,000 CV/g stated in claim 65. To the extent Chang suggests substituting niobium for tantalum powders, for sake of argument, that suggestion was made and illustrated for low BET surface area, low capacitance type powders.

Therefore, the Examiner's proposed substitution of niobium powder, based on the low capacitance powders taught by Chang, for the alleged high capacitance tantalum powder set forth in WO '248, would not yield a predictable result. In particular, it is not reasonably predictable that the Examiner's proposed combination of WO '248 and Chang would result in powder having a high capacitance of no less than 65,000 CV/g.

As indicated above, He et al. is not prior art to the claimed invention and for this reason alone it thus can not cure the above-indicated deficiencies of WO '248 and Chang with regard to claim 65. Furthermore, the deficiencies and problems with the Examiner's reliance on He et al., as set forth above, apply equally here.

In view of the above reasons, this rejection of claim 65 also should be reversed.

Conclusion

For the reasons set forth above, Appellants submit that the claims presently pending in the above-captioned application meet all of the requirements of patentability. It is therefore respectfully requested that the Honorable Board reverse the Examiner and remand this application for issue.

Respectfully submitted,

A handwritten signature in black ink, appearing to read "L. A. Kilyk", written in a cursive style.

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(10) Claims Appendix

36. Agglomerated niobium powder characterized by a capacitance of at least 65,000 CV/g and a DC leakage of less than 5.0 nA/CV when the agglomerated niobium powder is tested under electrical properties test conditions of sintering at a temperature of 1100° C for 10 minutes and anodizing at a formation voltage of 20Vf at 60° C.

37. The niobium powder of claim 36, wherein said capacitance is from 65,000 to about 150,000 CV/g.

38. The niobium powder of claim 36, wherein said capacitance is from about 75,000 to about 175,000 CV/g.

39. The niobium powder of claim 36, wherein said capacitance is from about 100,000 to about 250,000 CV/g.

40. The niobium powder of claim 36, wherein said capacitance is from about 125,000 to about 250,000 CV/g.

41. The niobium powder of claim 36, wherein said capacitance is from about 100,000 to about 210,000 CV/g.

42. The niobium powder of claim 36, wherein said DC leakage is from about 0.50 nA/CV to less than 5.0 nA/CV.

43. The niobium powder of claim 36, wherein said niobium powder comprises flaked niobium powder.

44. The niobium powder of claim 36, wherein said niobium powder has a BET surface area of at least about 5.5 m²/g.

45. The niobium powder of claim 36, wherein said niobium powder has a BET surface area of at least about $7.0 \text{ m}^2/\text{g}$.

46. The niobium powder of claim 36, wherein said niobium powder has a BET surface area of at least about $10 \text{ m}^2/\text{g}$.

47. The niobium powder of claim 36, wherein said niobium powder has a BET surface area of from $6.0 \text{ m}^2/\text{g}$ to about $12 \text{ m}^2/\text{g}$.

48. The niobium powder of claim 36, wherein said niobium powder has an oxygen content of less than 1,000 ppm.

49. The niobium powder of claim 36, wherein said niobium powder has an oxygen content of from about 2,000 ppm to about 60,000 ppm.

50. The niobium powder of claim 36, wherein said niobium powder is nitrogen doped.

51. The niobium powder of claim 36, wherein said niobium powder has at least about 100 ppm of nitrogen present.

52. The niobium powder of claim 36, wherein said niobium powder has nitrogen present in an amount of from about 100 ppm to about 5,000 ppm.

53. The niobium powder of claim 36, wherein said niobium powder has a flow of at least about 80 mg/s.

54. The niobium powder of claim 36, wherein said niobium powder has a flow of from about 80 to about 500 mg/s.

55. The niobium powder of claim 36, wherein said niobium powder has a Scott Density of about 35 g/in^3 or less.

56. The niobium powder of claim 36, wherein said niobium powder has a Scott Density of from about 10 to about 35 g/in^3 .

57. The niobium powder of claim 36, wherein said niobium powder has a particle size of from 5 to 80 microns.

58. The niobium powder of claim 36, wherein said niobium powder has an aspect ratio of from about 3 to about 300.

61. The niobium powder of claim 36, wherein said niobium powder has a Scott Density of about 35 g/in^3 or less, and a flow of at least about 80 mg/s.

62. The niobium powder of claim 61, wherein said niobium powder has a particle size of from 5 to 80 microns.

63. The niobium powder of claim 62, wherein said niobium powder has an aspect ratio of from about 3 to about 300.

64. The niobium powder of claim 62, wherein said niobium powder is agglomerated.

65. Agglomerated niobium powder characterized by a capacitance of at least 65,000 CV/g and a DC leakage of less than 5.0 nA/CV when the agglomerated niobium powder is tested under electrical properties test conditions of sintering at a temperature of 1100° C for 10 minutes and anodizing at a formation voltage of 35Vf at 60° C.

(11) Evidence Appendix

a) Appendix I: Declaration under 37 C.F.R. § 1.132 of Heather Enman (“the 2006 Enman Declaration,” filed with Amendment filed on February 24, 2006).

b) Appendix II: Declaration under 37 C.F.R. § 1.132 of Heather Enman (“the 2007 Enman Declaration,” filed with Amendment filed on April 30, 2007).

c) Appendix III: Declaration under 37 C.F.R. § 1.132 of Jonathon L. Kimmel (“Kimmel Declaration,” filed with Amendment filed on April 30, 2007).

d) Appendix IV: Brochure of Starck, ‘HCST -Tantalum-Niobium’ (“the Starck Brochure,” filed with Amendment of October 19, 2007).

APPENDIX I

THE 2006 ENMAN DECLARATION

I hereby certify that this correspondence is being facsimile transmitted to the United States Patent and Trademark Office, Fax No. 1-571-273-8300 on February 24, 2006.

Kim Blum
Name (Print)


Signature

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

| | | |
|---------------------------------------|---|-----------------------------|
| In re Application of: HABECKER et al. |) | Examiner: Daniel J. Jenkins |
| |) | |
| Application No.: 10/795,968 |) | Group Art Unit: 1742 |
| |) | |
| Filed: March 8, 2004 |) | Confirmation No.: 8631 |
| |) | |
| Docket No.: 99066CON2 (3600-198-02) |) | |

For: HIGH CAPACITANCE NIOBIUM POWDERS AND ELECTROLYTIC CAPACITOR ANODES

DECLARATION UNDER 37 C.F.R. § 1.132

Mail Stop AF
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

February 24, 2006

Sir:

I, Heather Enman, do declare and state as follows:

(1) I am currently employed with Cabot Corporation, and have been working in the niobium powder area, doing research and product development since January 2001.

(2) I have a bachelor's and master's degree in Chemical Engineering from Rutgers University.

(3) I am familiar with the Office Action dated September 26, 2005, received in the examination of U.S. Patent Application No. 10/795,968. I am also familiar with the cited references relied upon by the Examiner.

(4) In order to show that the powders of Chang (U.S. Patent No. 5,448,447), in particular, niobium powders having a BET around $0.55 \text{ m}^2/\text{g}$ would not have the capacitance set forth in claim 36 of the present application, experiments were uncovered to show the capacitance

U.S. Patent Application No. 10/795,968
Declaration Under 37 C.F.R. § 1.132
Reply to Office Action of September 26, 2005

achieved by similar powders under similar conditions. These experiments were done by me in the past, or under my direct supervision.

(5) As can be seen by the attached data, Table 1, a Nb powder having a BET of 0.58 m^2/g when formed at a formation voltage of 35 volts at a formation temperature of 60° C, and at a sinter temperature of 1,300° C, for a sinter time of 10 minutes, had a capacitance of 26,286 CV/g. Furthermore, as shown on the attached graph, the affects of sintering temperature can be extrapolated to show that a capacitance of 26,286 CV/g can be extrapolated to a capacitance of about 40,000 CV/g for a sinter temperature of 1,100°. In making this graph, I have assumed a linear relationship, which is a reasonable assumption, and the resulting capacitance for the 0.58 m^2/g BET sample would be about 40,000 CV/g at 1,100° C. This is significantly below the 65,000 CV/g stated in claim 36. Even if a fair degree of non-linearity exists in the CV/g--sintered temperature relationship, the capacitance for the 0.58 m^2/g BET sample would still be below 65,000 CV/g in my opinion based on my work in the niobium powder area.

(6) Accordingly, it is in my opinion that the powders of Chang, with respect to niobium powders, would not be capable of the electrical characteristics set forth on claim 36 of the present application.

(7) I hereby declare that all statement made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

U.S. Patent Application No. 10/795,968
Declaration Under 37 C.F.R. § 1.132
Reply to Office Action of September 26, 2005

2/20/06
Date

Heather Enman
Heather Enman

Attachment I: Experiments -- Graph

Niobium Metal Samples

| Notebook ID | BET (m ² /g) | Press Density (g/cc) | Formation Voltage | Formation Temp (C) | Sinter Temp (C) | Sinter Time (min) | CV/g | nA/CV |
|----------------|----------------------------|----------------------------|----------------------|-----------------------|--------------------|----------------------|-------|-------|
| 8656-85-E | 0.58 | 3.0 | 35 | 60 | 1400 | 10 | 19781 | 0.49 |
| 8656-85-E | 0.58 | 3.0 | 35 | 60 | 1300 | 10 | 26286 | 0.78 |
| 8656-89-270SH | 0.71 | 3.0 | 35 | 60 | 1300 | 10 | 26225 | 2.49 |
| 8656-89-270SAH | 0.65 | 3.0 | 35 | 60 | 1300 | 10 | 24038 | 0.98 |

Formation

Current Density 50mA/g
35V Ef @ 60 Deg C./0.1% H₃PO₄ Electrolyte

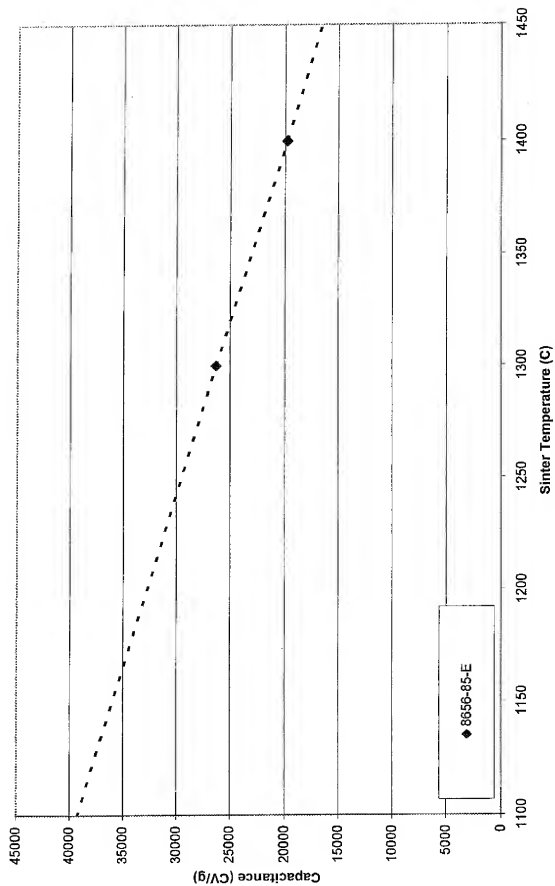
DC Leakage Testing

70% Ef (24.5 VDC) Test Voltage
60 second charge time
10% H₃PO₄ @ 21 Deg C.

Capacitance Testing:

18% H₂SO₄ @ 21 Deg C.
120 Hz

Figure 1: Capacitance Change with Sinter Temperature



APPENDIX II

THE 2007 ENMAN DECLARATION

I hereby certify that this correspondence is being facsimile transmitted to the United States Patent and Trademark Office, Fax No. 1-571-273-8300 on April 30, 2007.

Kim Blum
Name (Print)


Signature

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

| | | | | |
|-----------------------|-------------------------|---|-------------------|---------------------|
| In re Application of: | HABECKER et al. |) | Examiner: | Kathleen A. McNelis |
| Application No.: | 10/795,968 |) | Group Art Unit: | 1742 |
| Filed: | March 8, 2004 |) | Confirmation No.: | 8631 |
| Docket No.: | 99066CON2 (3600-198-02) |) | | |

For: HIGH CAPACITANCE NIOBIUM POWDERS AND ELECTROLYTIC CAPACITOR ANODES

DECLARATION UNDER 37 C.F.R. § 1.132

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

April 30, 2007

Sir:

I, Heather Enman, do declare and state as follows:

- (1) I am currently employed with Cabot Corporation, and have been working in the niobium powder area, doing research and product development since 2001.
- (2) I have a bachelor's degree and a master's degree in Chemical Engineering from Rutgers University.
- (3) I am familiar with the Office Actions dated September 26, 2005 and October 30, 2006, received in the examination of U.S. Patent Application No. 10/795,968. I am also familiar with the cited references relied upon by the Examiner.
- (4) In my previous Declaration dated February 24, 2006, the Examiner's position was that the tantalum powder of Chang could be niobium powder and, therefore, in my previous Declaration, it was shown that niobium powders having a BET around 0.55 m²/g would not have

the capacitance set forth in claim 36 of the present application.

(5) From the most recent Office Action dated October 30, 2006, it is my understanding the Examiner requested data from the standpoint of a formation voltage of 20 volts. As can be seen by the attached data, Table 1, a Nb powder having a BET of $0.58 \text{ m}^2/\text{g}$ when formed at a formation voltage of 35 volts, a formation temperature of 60°C , and at a sinter temperature of $1,300^\circ \text{C}$, for a sinter time of 10 minutes, had a capacitance of 26,286 CV/g. Using the data points in the attached Table, as shown on the attached graph, the affects of sintering temperature can then be extrapolated to show that a capacitance of 26,286 CV/g can be extrapolated to a capacitance of about 40,000 CV/g for a sinter temperature of $1,100^\circ$. Further, the same lot of niobium powder was tested at a formation voltage of 30 volts and 35 volts, as shown on the attached Table. From knowing the effects of various formation voltages for this powder, it was possible to determine a mathematical relationship such that a prediction could be made as to what the capacitance would be at a formation voltage of 20 volts, as shown on the attached Table and Figure. In addition, from this prediction, as was done in my earlier Declaration, a prediction was then made on what the capacitance would be at a formation voltage of 20 volts and a sintering temperature of 1100°C . In making this graph, I have assumed a linear relationship, which is a reasonable assumption, and the resulting capacitance for the $0.58 \text{ m}^2/\text{g}$ BET sample would be about 43,600 CV/g at $1,100^\circ \text{C}$ and a Vf of 20 volts. This is significantly below the 65,000 CV/g stated in claim 36. Even if a fair degree of non-linearity exists in the CV/g--sintered temperature relationship, the capacitance for the $0.58 \text{ m}^2/\text{g}$ BET sample would still be below 65,000 CV/g in my opinion based on my work in the niobium powder area.

(6) Furthermore, if the tantalum powder of Chang, which has a BET of less than $0.6 \text{ m}^2/\text{g}$, was tested for electrical properties at a temperature of 1100°C for 10 minutes and anodized using a formation voltage of 20 volts at 60°C , it is extremely unlikely that any anode would form in

order to determine the electrical properties of the tantalum powder due to the low surface area of the tantalum powder and, moreover, the DC leakage of such an anode, even if it was formed, would be greatly above 5.0 nA/CV. This is due to the low surface area of the tantalum and that tantalum is not the same as niobium, even when the surface areas are similar. A tantalum powder run at these test conditions would essentially be considered a near-failure or failure.

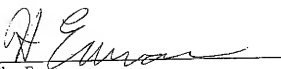
(7) Accordingly, it is in my opinion that the powders of Chang, with respect to niobium powders, would not be capable of the electrical characteristics set forth on claim 36 of the present application.

(8) I hereby declare that all statement made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date

4/30/07

Heather Enman



Attachment I: Experiments -- Graph

Niobium Metal Samples

| Sample ID | BET (m ² /g) | Press Density (g/cc) | Formation Voltage | Formation Temp (C) | Sinter Temp (C) | Sinter Time (min) | CV/g | nA/CV |
|--|-------------------------|----------------------|-------------------|--------------------|-----------------|-------------------|-------|-------|
| 8656-85-E | 0.58 | 3.0 | 25 | 60 | 1300 | 10 | 26463 | 0.51 |
| 8656-85-E | 0.58 | 3.0 | 30 | 60 | 1300 | 10 | 26452 | 0.43 |
| 8656-85-E | 0.58 | 3.0 | 35 | 60 | 1300 | 10 | 26286 | 0.78 |
| 8656-85-E Predicted | 0.58 | 3.0 | 20 | 60 | 1300 | 10 | 26577 | |
| Predicted from cap slope with temperature for 20Vf | | | | | | | | |
| 8656-85-E | 0.58 | 3.0 | 20 | 60 | 1100 | 10 | 43619 | |
| 8656-85-E | 0.58 | 3.0 | 35 | 60 | 1100 | 10 | 39296 | |

CV/g at 20Vf and 1300G sinter is predicted by the equation $y = -17.7(x) + 26531$

CV/g at 35Vf and 1100C sinter is predicted by the equation $y = -65.05(x) + 110851$

Cap change from 35Vf to 20Vf is an increase of approximately 11%. Therefore CV/g at 20Vf and 1100C for this powder is

$$CV/g = 43619 = 39296(0.11) + 39296$$

Formation

Current Density 50mA/g

35V Ef @ 60 Deg C./0.1% H3PO4 Electrolyte

DC Leakage Testing

70% Ef (24.5 VDC) Test Voltage

60 second charge time

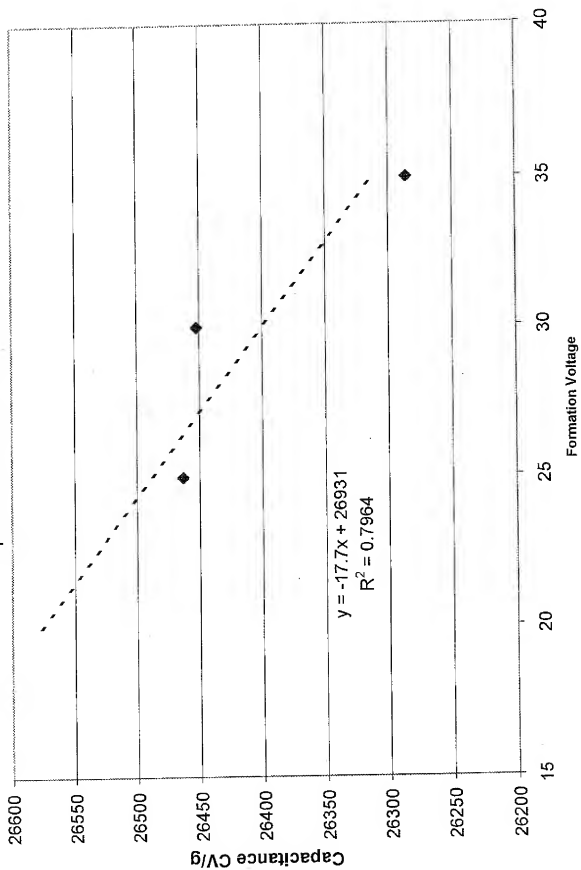
10% H3PO4 @ 21 Deg C.

Capacitance Testing:

18% H2SO4 @ 21 Deg C.

120 Hz

Capacitance Profile for 0.58 BET Nb



APPENDIX III

THE KIMMEL DECLARATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: FIFE, *et al.*

Application No.: 09/632,714

Filed: August 4, 2000

Group Art Unit: 1775

Examiner: S. STEIN

For: NIOBIUM POWDERS AND NIOBIUM ELECTROLYTIC CAPACITORS

DECLARATION OF JONATHAN L. KIMMELAssistant Commissioner for Patents
Washington, D.C. 20231

May 21, 2001

Sir:

I, Jonathon L. Kimmel, declare and state as follows:

1. I am currently an employee of Cabot Corporation, the Assignee of the above-identified patent application. Furthermore, my responsibilities at Cabot Corporation include being Manager of the research and development of niobium based products including the invention set forth in the above-identified patent application. I have been employed by Cabot Corporation since February 1, 1998. Furthermore, my education background includes holding a Bachelor's Degree in electrical engineering and doing prior work with such companies as DuPont Corporation.

2. I am familiar with the invention and the claims set forth in the above-identified patent application as well as the Office Action dated January 19, 2001. Furthermore, I am familiar with the documents referenced by the Examiner including Chang, Schiele, and Fry.

3. From the Office Action, it is my understanding that the Examiner is arguing that tantalum and niobium are similar such that substitution of each powder is possible.

4. In response to the Examiner's comments set forth in the Office Action, it is my opinion, which is based on experience and knowledge in this area, that niobium can not simply be substituted for tantalum especially in the electrode capacitor area.

Declaration of Jonathon L. Kimmell

5. To further support this point, attached is information from the Tantalum-Niobium International Study Center (also known as TIC) that further explains the current applications for tantalum and niobium. While all the applications may not be mentioned, it is important to realize that the first application mentioned for tantalum is capacitors. However, there is no mention under the niobium section for capacitors. This further confirms that niobium and tantalum are not easily substituted contrary to the Examiner's reasoning. In addition, at page 3 of the TIC printout, there is a history of tantalum and niobium identified as "Tantalum And Niobium-Dissimilar Twins?" While this section does not fully explain the differences between tantalum and niobium, it is interesting to note that this brief article appreciates the significant differences between tantalum and niobium.

6. In addition, attached are graphs related to worldwide capacitor consumption which are from the TIC and are publicly available. As can be seen, from the bar graphs, the production of niobium was so insignificant for capacitor consumption that it did not even record on the bar chart for 1990. This is even true for 2000. Similarly, in the table that shows worldwide capacitor consumption for 1995 and 2000, again, it can clearly be seen that the consumption for niobium was non-existent. Thus, it is clear that niobium even as of 1990 and as of the filing date of the present application was not used commercially in capacitor manufacturing. Thus, if niobium was an easy substitute for tantalum, manufacturers would be using niobium because it is less expensive and is more available. However, manufacturers are not using niobium in the capacitor market. It is clear that niobium and tantalum are different and niobium is not a substitute for tantalum capacitors. One reason is because the pentoxide layer formed on the niobium anode readily becomes unstable for the life of the capacitor thus resulting in the pentoxide film becoming less stable and causing a breakdown of the capacitor. This has been shown by accelerated life testing of the previous niobium

Declaration of Jonathon L. Kimmell

capacitor's problem. This problem does not occur in tantalum capacitors. To overcome this problem, the present invention uses high surface area niobium and preferably uses various dopants such as oxygen and the like.

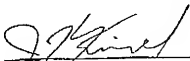
7. In addition, as set forth in the beginning of the present application, niobium has never been considered as a substitute for tantalum by any commercial industry or manufacturer of the powders. This is, as stated above, confirmed by the charts that show capacitor consumption which has not included niobium. Furthermore, according to the Examiner, if tantalum and niobium are so similar, why is the DC leakage different at formation voltages above 60 volts? This is confirmed by the attached charts comparing niobium with tantalum with respect to various formation voltages in DC leakage. As can be seen, the DC leakage for niobium rapidly increases at formation voltages above 50 volts which is quite different from tantalum powders. In addition, the capacitance capabilities of niobium versus tantalum are quite different as well as the effect of sintering temperatures for DC leakage. This is reflected in the attached charts which show these differences with respect to capacitance based on formation voltages.

8. Thus, as indicated by the various evidence set forth above, niobium is a separate product from tantalum and niobium has different properties from tantalum with respect to the capacitance achieved, the DC leakage, the crush strength, and other properties related to using niobium in the formation of an anode when compared to tantalum being used in the formation of an anode. Thus, there is not simple interchangeability of niobium for tantalum.

Declaration of Jonathon L. Kimmell

I hereby declare that all statement made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

5/21/01
Date


Jonathon L. Kimmell

The metals

TANTALUM



Some current applications:

1. As metal powder for the production of tantalum capacitors (dependent on the insulating property of the tantalum oxide films) and as metal wire for their connection to circuits. This biggest single use depends on the large surface area of the finely divided powder, and on the purity of the metal.
2. As fabricated metal for the construction of chemical process plant, and equipment such as heat exchangers, due to its resistance to corrosion.
3. As an ingredient of superalloys, principally for use in aircraft engines and spacecraft.
4. As the carbide, in hard metal cutting tools, also with tungsten carbide and cobalt binders.

The first item accounts for about 50% of current tantalum usage.

NIOBIUM



Some current applications:

1. In structural steels (especially high strength low alloy (HSLA) steels with up to 1% Nb). In heat resisting steels and superalloys (iron, nickel or cobalt based).
2. As an alloy with titanium or tin for superconductive magnets.
3. As the oxide (Nb_2O_5) in high refractive index glass, as lead niobate in piezoelectric devices, and as single crystal lithium niobate in surface acoustic wave filters for television sets and similar equipment.
4. With copper in powder metallurgy composites for components requiring high strength with conductivity.
5. As an alloying addition to zirconium for nuclear reactor fuel tubes.

6. As the carbide, in cutting tools, with tungsten and tantalum carbides.
7. As the fabricated metal, for chemical plant, and, alloyed with zirconium or other metals, for high temperature applications in aerospace.

The first item accounts for about 90% of current niobium usage

The association: T.I.C.

Tantalum-Niobium International Study Center,
40 rue Washington,
1050 Brussels,
Belgium.
Telephone: +32 2 649 51 58
Fax: +32 2 649 64 47
e-mail: tantniob@agoronet.be

The Tantalum-Niobium International Study Center is an international, non-profit association founded under Belgian law. It received Royal Assent in 1974, and has its headquarters in Brussels, Belgium. It has more than 50 members, which are companies involved in raw material production, processing, assaying, trading, capacitor manufacture, chemical process equipment manufacture, and are situated in more than 20 different countries across the world. A list of the current members is included in this site, with an indication of the main field of activity of each. The association is managed by an Executive Committee with members drawn from all parts of the industry and regions of the world.

Alms

The main goal of the T.I.C. is to increase awareness of tantalum and niobium and their remarkable properties, and to promote the use of the metals and their products. The association organises each October a conference with technical and business presentations, and the programme also includes a plant tour to a factory or mine associated with the production or use of these metals, sponsored by one of the members. At intervals the T.I.C. also organises an international Symposium of about three days, and such a meeting is planned for the west coast of the United States in October 2000. A quarterly Bulletin with some of the presented papers and other articles of general interest to the industry is published; copies may be requested from the Secretariat. The association also collects statistics on the industry, these are supplied to the members, who also receive a monthly set of abstracts and news headlines.

Membership

Companies which have a continuing interest in one or both of the metals are eligible to apply for membership. Information and application forms may be obtained from the Secretariat.

Associate membership is available to individuals and to non-profit organisations with an interest in the metals which is not commercial, such as researchers, academics, authors.

Tantalum and Niobium — Dissimilar Twins?

In 1801 an American chemist named Hatchett was testing a heavy black mineral from Connecticut and discovered that it contained a new element which he named 'Columbium'. A year later Eckberg in Sweden discovered two minerals each containing an oxide of an unknown element. This proved very difficult to dissolve in acids and frustrating to work with, so he named it 'Tantalum' after the Greek god Tantalus (who could not reach the water to drink or the apple to eat)

In 1844 the chemist Rose showed that another element was present in the Swedish mineral, and he named it 'Niobium' after Niobe, the daughter of Tantalus. Only in 1866 did Marignac develop a method of separating the two elements chemically by taking advantage of the difference in solubility of the two potassium double fluorides (a procedure used until quite recently in the manufacture of the metals). The European 'Niobium' was soon shown to be identical to the American 'Columbium' and for nearly a century arguments raged over which name had priority. Finally in 1950 the international chemical body, by a majority decision, settled for niobium but the old name columbium is still in common use in the Americas, complicated by the fact that one of the two most common minerals of niobium is universally known as 'Columbite'. Of the two elements, niobium is far more abundant in the earth's crust than tantalum, nevertheless they almost always occur together. This results from the great chemical similarity of their oxides (which gave those early chemists so much trouble), and from their very similar atomic radii, so that they freely replace each other in minerals. The columbite mentioned above is an iron or manganese niobate and there is an iron manganese tantalate known as tantalite. A full range of mixtures between the extremes exists, all naturally occurring.

When the two elements were finally separated, and the metals produced, it was obvious that the similarity did not extend to all their physical properties. Niobium metal is very similar in density to iron, but tantalum is nearly twice as heavy. As a result of all these factors, and the relative abundance (and cheapness) of niobium, they have very different applications, but in some uses, in particular of the high purity metal and its alloys, there is some overlap. Dissimilar twins indeed!

Applications of tantalum and niobium are based on their ability to form a non-conducting layer of oxide on the surface of the metal, a dense, stable and adhesive layer of pentoxide.

Tantalum — Raw Materials and Processing

Until recently the majority of the world's production of tantalum was from the discard slags of tin smelters. The tin mineral cassiterite is frequently associated with columbite/tantalite ore, especially in Thailand, Australia, Brazil and Central Africa, and to a smaller extent in Malaysia. When the tin concentrates are smelted, the tin is reduced to metal, but the tantalum reports unreduced in the slag, from which it can be recovered by electric smelting and/or chemical extraction. Tin slags, particularly those from the Thaisarco smelter in Phuket, Thailand, used to be an important supplier of tantalum, but the decline of the tin industry in South East Asia since 1985 has meant the replacement of much of that source of tantalum by primary

Niobium — Raw Materials and Processing

A small part of industry's needs for niobium is recovered during the treatment of tin slags and columbite/tantalite minerals (mined in West Africa, Brazil, South East Asia and Australia) which are the source of much of the world's tantalum. However, more than 90% of all niobium is recovered as ferroniobium for use in steel-making, from the smelting (by reduction with aluminium) of the mineral pyrochlore, a calcium fluoniobate (which, when the niobium is replaced by tantalum, is known as microlite, and mined for tantalum). Two mines in Brazil (the CBMM mine at Araxá, and the Anglo-American mine at Catalão in Goiás) account for 80% of all niobium, and one in Canada (Niobec at St Honoré) for more than half the remainder.

mining of tantalite.

There are a number of mines now operating, two of the largest are open-cut operations in Western Australia (Greenbushes and Wodgina). A minor source resulted from a boom in tantalum prices in 1979/80 which caused the excavation of very large tonnages of old tantalum-bearing tin slags in South East Asia, where they had been used as ground fill since early this century. Some of these are still supplying the processors' needs but it is generally considered that stocks of them are now small. There is also a sizeable amount (about 25%) of recycling of scrap metal and of compounds of tantalum.

Tantalum and niobium are extracted from their ores, after concentration, by chemical means rather than by smelting. The concentrates are attacked by $\text{HF}/\text{H}_2\text{SO}_4$ which brings the tantalum and niobium compounds into solution. The acid solution is mixed thoroughly with MIBK (methyl-iso-butyl ketone) which dissolves the tantalum and niobium compounds into the ketone while leaving impurities in the aqueous solution. The organic and inorganic solutions form separate layers and the organic (ketone) solution can be separated from the aqueous layer (liquid-liquid separation). The niobium is then stripped with dilute acid, and the tantalum subsequently extracted by acid ammonium fluoride. For tantalum, the metal is usually produced in powder form by sodium reduction of the fluoride. It can then be compacted (as it is for capacitors) to final shape, or may be melted (and refined) in an electron beam furnace.

Tantalum — Applications

For a brief period early this century, tantalum had a use in wire form as lamp filament, but the development of cheap tungsten wire put paid to this.

The biggest single use now of tantalum (accounting for 50% of the total) is as the powdered metal, mostly for the production of capacitors. These depend on the dielectric properties of a tantalum oxide film on a tantalum compact, but the amount of tantalum required for a particular service is reduced as the metal purity is raised. Technical advances in tantalum manufacture and increasing knowledge of the effect of other parameters such as particle size and shape have enabled capacitor makers to make large increases in their production of units with little significant

Pure niobium and its oxide are prepared through a chemical route similar to that applied to tantalum.



Niobium — Applications

The ferro-niobium which makes up most of niobium consumption is used in the preparation of steels, in particular those of the HSLA (high strength low alloy) type. Until recently the best-known use of the niobium-bearing steel has been in the manufacture of natural gas pipelines: the niobium gives the steel the strength to resist the great range of temperatures to which such pipes are subject. Niobium addition is now growing in other steels such as stainless, and this has been helped by the stability of its price compared with that of competing alloying elements.

Pure niobium, like tantalum, has considerable attraction as a material of construction for chemical plant, and in the formulation of superalloys, but it has an additional use with

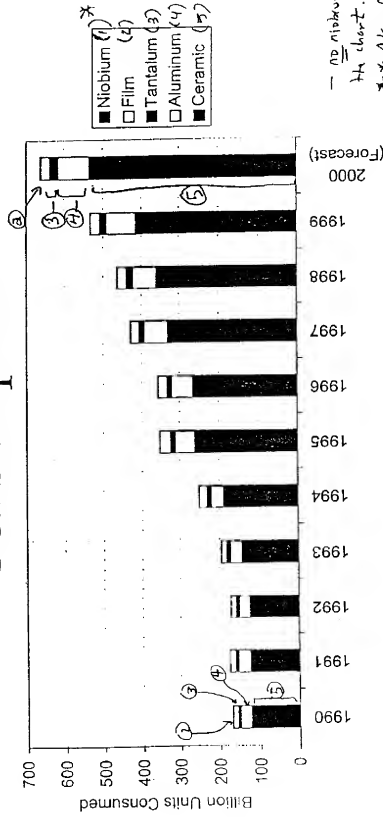
Increase in the weight of tantalum consumed. Also important as applications of tantalum are tantalum carbide used in cutting tools (with titanium and tungsten carbides), and pure or alloyed tantalum metal, much of it for corrosion or heat resistant chemical plant equipment, or in superalloys for jet engines



alloyed with titanium or tin, as a superconductor. Superconducting applications include magnetic resonance imaging for medical diagnostics and in particle accelerators for physics research. The Mag-Lev train may be a more conspicuous use of this material in the future. The high strength magnetic fields required can be generated in a small volume by superconducting coils — in medicine, the technique is much safer than that using X-rays.

Niobium also has several non-metallic applications. An important one is in optical glass: when the silica in glass is replaced by niobium oxide, the refractive index is much greater, so thinner and lighter lenses can be used for the same focal length. This is already in use in many camera lenses and for eyeglasses.

Worldwide Capacitor Consumption



- no niobium made
the chart.
* * Also, for all other
bars - the same
order is followed.

Sources: Tantalum-Niobium International Study Center (TIC);
Paumonok Publications; Business Process Engineering(BPR)

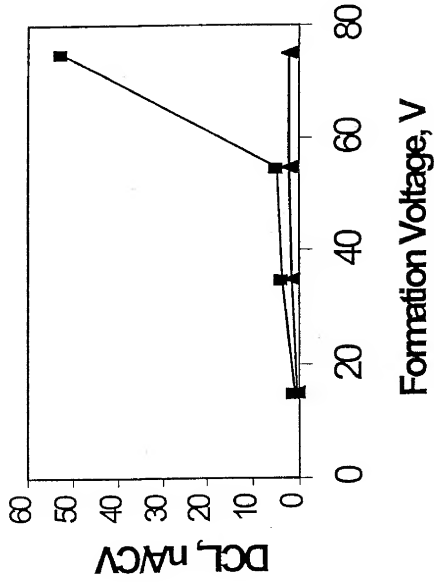
Worldwide Capacitor Consumption

| | 1995 | 2000 |
|--------------------------------|------------|------------|
| WW Consumption (Billion Units) | | |
| MLCC | 180 | 350 |
| SLC | 65 | 80 |
| Aluminum | 80 | 120 |
| DC Film | 28 | 34 |
| Tantalum | 14 | 25 |
| Other | 1 | 1 |
| Total | 368 | 610 |

Source: Tantalum-Niobium International Study Center (TIC)
as reported in Electronic Buyers' News 25-Jan-99, p36

DCL vs Formation Voltage

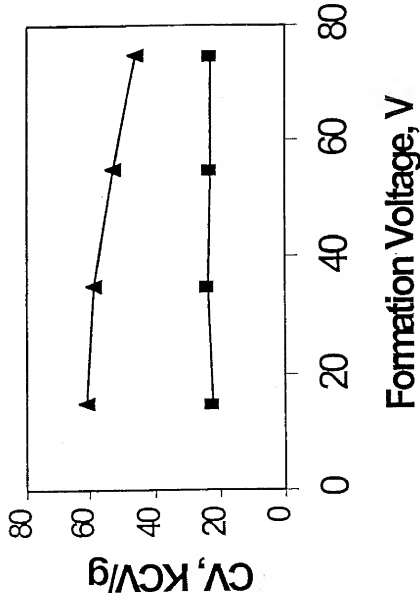
1300 Deg Sinter



—■— Nb —▲— Ta-C606

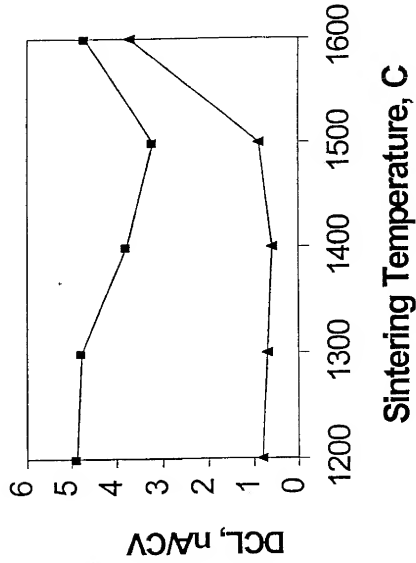
CV vs Formation Voltage

1300 Deg. Sinter



—■— Nb —▲— Ta-C806

DCL vs Sintering Temperature



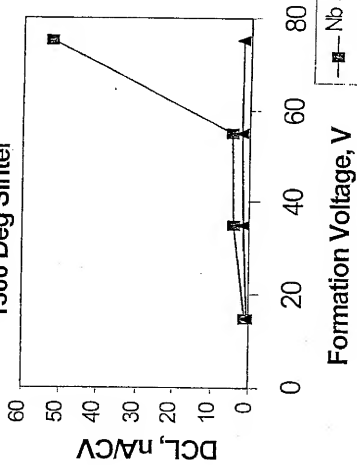
■ Nb —▲ Ta-C606

Niobium Vs. Tantalum

Forming Voltage Range

DCL vs Formation Voltage

1300 Deg Sinter



Experiment

Experiment:

Process*

Product*

Product Category*

7007-R51000

Performance Characteristics*

Responsibility: Dan White

Date: 03/21/2000

Title:

Exp# 2452 (c) Nb vs Ta C606 Anodization Voltage/85C Evaluation

Objectives:

Procedures:

[1] Samples:

- (a) 8171-33-C 1300 sintered anodes from Exp# 2452(a)
- (b) C606-C-144577 1300 sintered anodes from Exp# 2452(a)

[2] 15V Ef Anodization:

- (1) One Formation
 - N = 8 anodes per sample
 - (3) samples * (1) sinter = 3 trees + lab standard
- (2) Electrolyte; (.1% H3PO4 @ 85 C Deg 4.3mmho)
- (3) Constant current density: (135 ma/g)
- (4) Terminal Voltage = 15.0 VDC +/- 0.03
- (5) Terminal Voltage Time = 180 min -0/+5 min
- (6) 60C soak for 30 min
- (7) 60 C oven for 120 min

[3] DC Leakage/Capacitance-ESR Testing:

- (a) DC Leakage Testing —
 - 70% Ef Test Voltage
 - 60 and 120 second charge time
 - 10% H3PO4 @ 21 Deg C
- (b) Capacitance-DF Testing:
 - 18% H2SO4 @ 21 Deg C
 - 120 Hz

[4] 35V Ef Anodization:

- (1) One Formation
 - N = 8 anodes per sample
 - (3) samples * (1) sinter = 3 trees + lab standard
- (2) Electrolyte; (.1% H3PO4 @ 85 C Deg, 4.3 mmho)
- (3) Constant current density: (135 ma/g)
- (4) Terminal Voltage = 35.0 VDC +/- 0.03
- (5) Terminal Voltage Time = 180 min -0/+5 min
- (6) 60C soak for 30 min
- (7) 60 C oven for 120 min

[5] DC Leakage/Capacitance-ESR Testing:

- (a) DC Leakage Testing —
 - 70% Ef Test Voltage
 - 60 and 120 second charge time

- 10% H₃PO₄ @ 21 Deg C
(b) Capacitance-DF Testing:
18% H₂SO₄ @ 21 Deg C
120 Hz

[6] 55V Ef Anodization:

- (1) One Formation
N = 8 anodes per sample
(3) samples * (1) sinter = 3 trees + lab standard
- (2) Electrolyte; (.1% H₃PO₄ @ 85 C Deg 4.3 mmho)
- (3) Constant current density: (135 ma/g)
- (4) Terminal Voltage = 55.0 VDC +/- 0.03
- (5) Terminal Voltage Time = 180 min -0/+5 min
- (6) 60C soak for 30 min
- (7) 60 C oven for 120 min

[7] DC Leakage/Capacitance-ESR Testing:

- (a) DC Leakage Testing ---
70% Ef Test Voltage
60 and 120 second charge time
10% H₃PO₄ @ 21 Deg C
- (b) Capacitance-DF Testing:
18% H₂SO₄ @ 21 Deg C
120 Hz

[8] 75V Ef Anodization:

- (1) One Formation
N = 8 anodes per sample
(3) samples * (1) sinter = 3 trees + lab standard
- (2) Electrolyte; (.1% H₃PO₄ @ 85 C Deg 4.3 mmho)
- (3) Constant current density: (135 ma/g)
- (4) Terminal Voltage = 75.0 VDC +/- 0.03
- (5) Terminal Voltage Time = 180 min -0/+5 min
- (6) 60C soak for 30 min
- (7) 60 C oven for 120 min

[9] DC Leakage/Capacitance-ESR Testing:

- (a) DC Leakage Testing ---
70% Ef Test Voltage
60 and 120 second charge time
10% H₃PO₄ @ 21 Deg C
- (b) Capacitance-DF Testing:
18% H₂SO₄ @ 21 Deg C
120 Hz

[10] 100V Ef Anodization (After review of 75V Evaluation):

- (1) One Formation
N = 8 anodes per sample
(3) samples * (1) sinter = 3 trees + lab standard
- (2) Electrolyte; (.1% H₃PO₄ @ 85 C Deg 4.3mmho)
- (3) Constant current density: (135 ma/g)
- (4) Terminal Voltage = 100.0 VDC +/- 0.03
- (5) Terminal Voltage Time = 180 min -0/+5 min
- (6) 60C soak for 30 min
- (7) 60 C oven for 120 min

I11 DC Leakage/Capacitance-ESR Testing:

(a) DC Leakage Testing —

70% Ef Test Voltage

60 and 120 second charge time

10% H₃PO₄ @ 21 Deg C

(b) Capacitance-DF Testing:

18% H₂SO₄ @ 21 Deg C

120 Hz

APPENDIX IV

THE STARCK BROCHURE

HCST

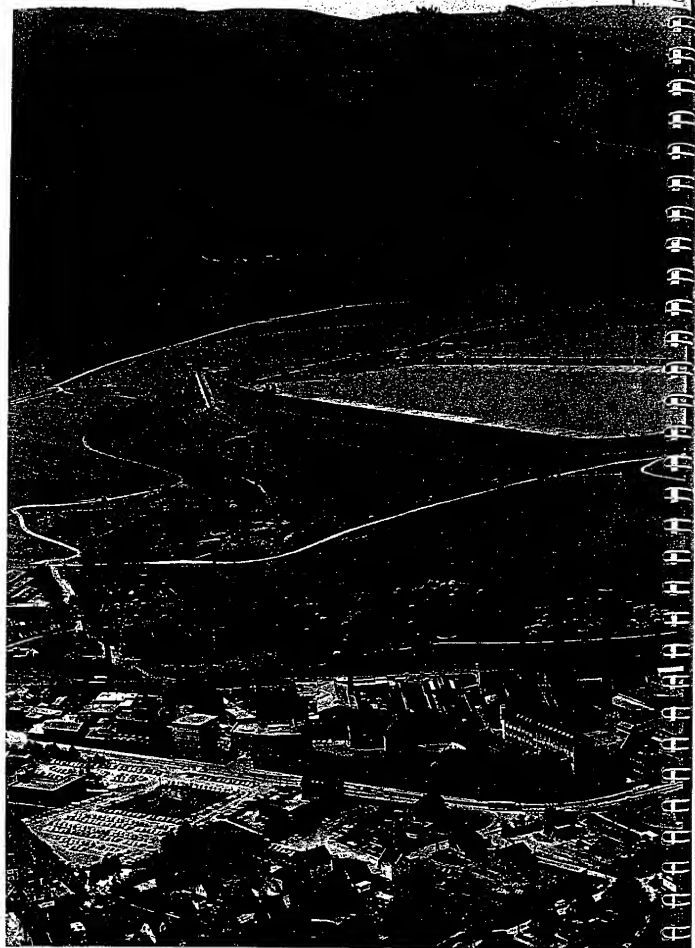
L. H. BELZ
MAY '85

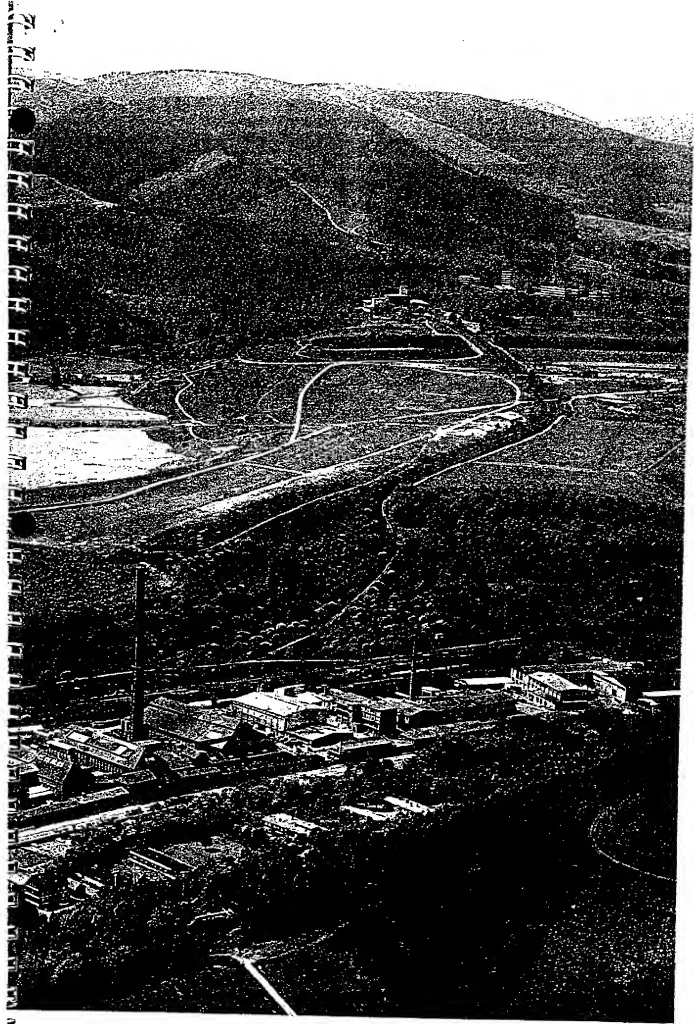
Tantalum
Niobium

Metallurgical

Remarks

General





General

Remarks

Quality Control

PL Series

Project Status

Analysis

Metallurgical

Tantalum by HCST

HCST known world-wide for its high quality metallurgical products, has been involved with the production of tantalum since 1949.

Since this time **HCST** has developed into one of the most important and experienced producers of tantalum in the world.

The development of the past years has shown that a sound raw material policy is the most important pre-condition for a tantalum production.

Therefore from the beginning **HCST** attached great importance to perform the tantalum extraction process in its own works starting from the raw material.

HCST therefore has designed and built extraction and separation plants for tantalum/niobium which permit great flexibility in operation by using wet chemical extraction or chlorination techniques.

This flexibility of operation permits the use of high grade tantalum ores such as tantalite and microlite with tantalum contents up to 70% as well as columbite and low grade by-products from other processes such as tin slags.

Besides **HCST** provides two ways to assure the supply of tantalum:

First, by developing new processes for recovering tantalum ores with lower and lower Ta-contents and secondly by developing Ta-powders for the capacitor industry with higher and higher capacitance thus insuring better utilization of the tantalum still available. (Powder type PL-18000 R today represents world-wide the powder with the highest yield of capacitance)

The research and development department of **HCST** is working very intensively in this field today.

The processes which are common practice at **HCST** are based on the application of the following **HCST** patents and the following pending patents:

DE-PS 2133104
DE-PS 2517180 ✓
DE-PS 2537354
DE-PS 2610224

DE-OS 2733193
DE-OS 3005207
DE-OS 3113335
DE-OS 3130392

or of the corresponding foreign patents.

This catalogue deals with the tantalum and niobium products offered by **HCST**. Special importance is being attached to the powder grades used for the production of tantalum capacitors.

Tantalum and Niobium Products by HCST

The following items are included in our list of tantalum "Capacitor Grade" products:

1. Tantalum powder -- "Capacitor Grade"
Series PL: Powders of high to highest capacitance for medium working voltage.
Series 600: Powders of high purity of low to high capacitance for high to highest working voltage.
2. Sintered tantalum anodes for electrolytic capacitors.
3. Niobium powder "Capacitor Grade" (on request).

Other tantalum and niobium products manufactured by **HCST** include:

5. Tantalum metal "Metallurgical Grade".
 - 5.1 Tantalum metal powder for powder metallurgy.
 - 5.2 Tantalum metal powder and pellets for melting.
 - 5.3 Tantalum scrap.
6. Tantalum for special purposes.
 - 6.1 Tantalum powder "Spraying Grade".
 - 6.2 Tantalum powder "Sub-Micron-Powder" (on request).
 - 6.3 Tantalum powder with especially high purity for scientific purpose.
7. Tantalum electron beam melted ingots.
8. Tantalum carbide, niobium carbide, tantalum-niobium carbide and tantalum-bearing triple and tetra carbides for the cemented carbide industry.
9. Tantalum oxide, niobium oxide and tantalum-niobium oxide chemical pure and of highest purity for the optical and ceramic industry. This includes the application for ceramic capacitors also.
10. Tantalum chloride, niobium chloride for surface coatings and other applications.
11. Potassium tantalum fluoride.

Should you be interested in these products, please ask for further informations.

Capacitor Grades

Metallurgical Grades

**Tantalum Powder Capacitor Grade
Grade PL-8000**

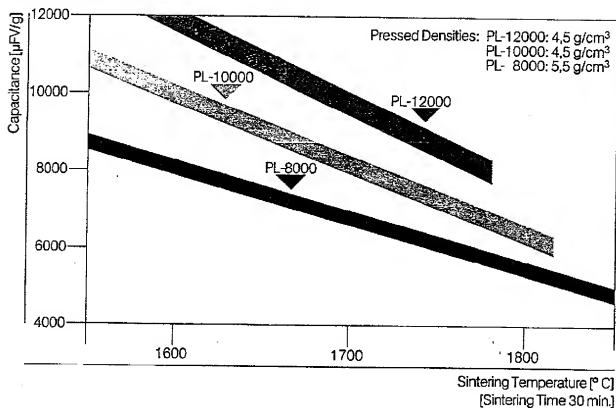
PL-8000

PL-8000 represents the powder with the lowest yield of capacitance per gram and the highest working voltage in the PL-series. It has a good flowability and permits pressed densities down to 5.0 g/cm³. PL-8000 is recommended especially for the production of medium voltage 35 V capacitors.

Recommended conditions for processing

Sintering temperature: 1600 – 1850°C
Sintering time: 15 – 30 minutes
Pressed density: 5,0 – 6,0 g/cm³
can also be pressed without binder
Yield of capacitance: 5000 – 8000 $\mu\text{FV/g}$ (Wet test)
Working voltage: up to 35 V

Typical Capacitance



Typical Electrical Characteristics

| Grade | $\mu\text{FV/g}$ | $\mu\text{FV/cm}^3$ | Sintering Temperature (°C) | VBD (V) | DCL (nA/ μFV) |
|---------|------------------|---------------------|----------------------------|---------|---------------------------|
| PL-8000 | 8000 | 47500 | 1600 | 180 | 0,4 |
| PL-8000 | 6800 | 44000 | 1700 | 200 | 0,30 |
| PL-8000 | 5500 | 38500 | 1800 | 220 | 0,2 |

Anode weight: 0,4 g, Pressed density: 5,5 g/cm³
Sintering time: 30 min, Formation voltage: 100 V

Series 600 (900)

660, 690-E, 690-S and 900-HC are the so-called "electron beam melted" powders. Their low contamination with impurities provides a high reliability at the highest working voltages.

The powders of this series are showing the following properties which are important for processing:

1. They can be pressed without binder
2. They show an excellent flowability
3. The high strength of the agglomerates guarantees that the excess powder does not show any significant change in processing.

All the characteristics, such as capacitance per gram, capacitance per volume of the two powder series, are shown on the following tables.

In the attached data sheets you will find further particulars of the powders discussed.

Capacitance per Gram

| Type \ $\mu\text{FV/g}$ | 1000 | 5000 | 10000 | 15000 | 20000 |
|-------------------------|------|------|-------|-------|-------|
| PL-18000R | | | | | |
| PL-12000 | | | | | |
| PL-10000 | | | | | |
| PL-8000 | | | | | |
| 900-HC | | | | | |
| 690-S | | | | | |
| 690-E | | | | | |
| 660 | | | | | |

Capacitance per Volume

| Type \ $\mu\text{FV/cm}^3$ | 10000 | 50000 | 100000 |
|----------------------------|-------|-------|--------|
| PL-18000R | | | |
| PL-12000 | | | |
| PL-10000 | | | |
| PL-8000 | | | |
| 900-HC | | | |
| 690-S | | | |
| 690-E | | | |
| 660 | | | |

Quality Control

A considerable amount of highly skilled analytical effort is employed to ensure consistently high quality in our tantalum products. This applies even to the starting materials such as the ores and the chemicals used to process them.

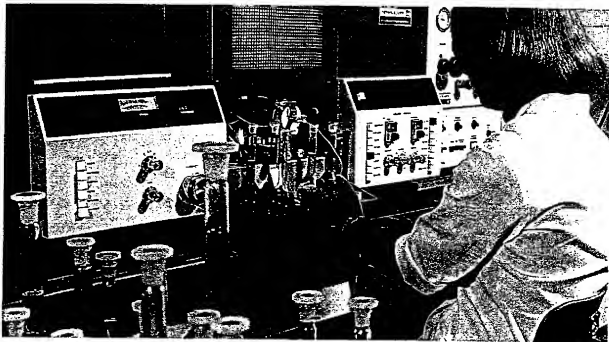
Every step in the processing is carefully controlled, especially in the stages liable to have influence on the electrical, physical and chemical properties of the final product.

Among the methods we use are:

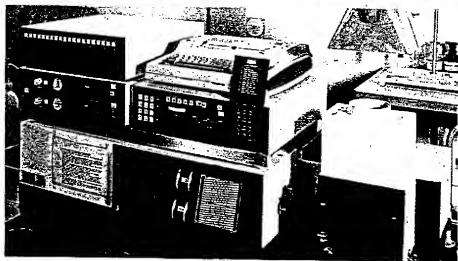
Determination of Metallic Contaminations

| | |
|-----------------------------|--------------------------------------|
| Emission spectroscopy | (3,5 m RSV, Plan-gitterspectrograph) |
| Spectrophotometry | (Spectronic 88, Bausch & Lomb) |
| X-ray fluorescence analysis | (Philips PW 1450/PW 1410) |
| Atom absorption | (Perkin-Elmer 503) |

Plasma Spectrometer (DC-Argon Plasma Echelle Spectrometer Spectrometrics Plasma Spectraspan II)



Atomic absorption spectrometer.



Plasma spectrometer

Determination of the Gas Content (Oxygen, Hydrogen, Nitrogen)

Vacuum fusion

(Leybold-Heraeus VH 9,
Gas Evolograph 0583 St. Re
(Leco TC 136))

Inert-carrier-gas fusion

(Leco TC 136)

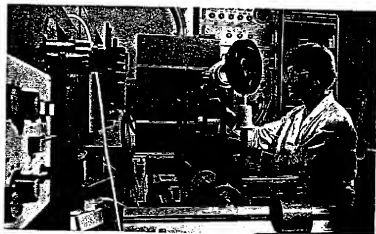
Determination of Carbon

Coulometric analysis

(Schoeps apparatus CTA 5 C)



*Inert-carrier-gas fusion
(Leco TC 136)*



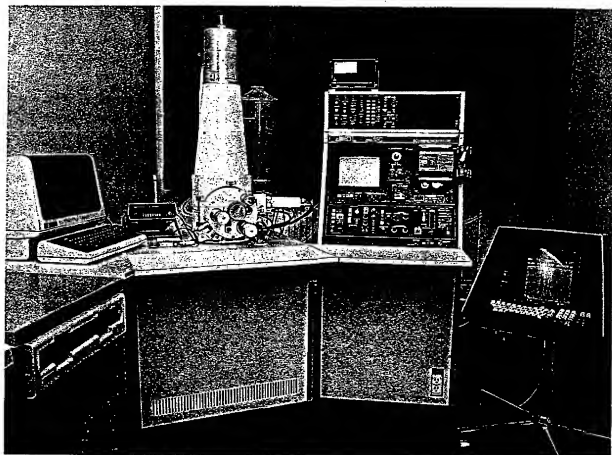
*3.5-m plane grating spectrophotograph for
photographic and photoelectric recording,
combined with an arc-spark stand and a
glow discharge lamp.*

Physical Properties

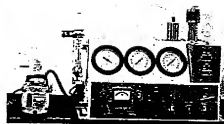
| | |
|-------------------------------|---|
| Scott density | (Scott, ASTM: B 329 - 76) |
| Average particle size | (Fisher Sub-Sieve Sizer, ASTM: B 330 - 82) |
| Screen distribution | (DIN or ASTM: B 214 - 76, E 11) |
| Flow properties | (Hall-Flow-Index, ASTM: B 213 - 77) |
| Particle size distribution | (Sedimentation: WAB, ASTM: B 430 - 79) Roller Analyzer, ASTM: 293 - 76) |

In addition these are several methods for determining further physical properties by means of

| | |
|--|--|
| Scanning Electron Microscope | (Jeol JSM-T100, JSM-35 C) |
| Mercury Porosimetry and BET method specific surface Sedigraph | (DIN 66132) (5000 D, Micromeritics) |



Scanning electron microscope with energy dispersive micro-analysis



Mercury porosimeter

Test Conditions (Wet Test)

| | 270 V | 200 V | 100 V (70 V) |
|------------------------------|--|-----------------------------------|-----------------------------------|
| Formation conditions: | | | |
| Electrolyte | 0,01 wt-% H_3PO_4 | 0,01 wt-% H_3PO_4 | 0,01 wt-% H_3PO_4 |
| Temperature | $90 \pm 2^\circ\text{C}$ | $90 \pm 2^\circ\text{C}$ | $90 \pm 2^\circ\text{C}$ |
| Formation voltage | 270 V | 200 V | 100 V (70 V) |
| Final voltage held for | 120 min | 120 min | 120 min |
| Formation Current | 35 mA/g (0–200 V) 12 mA/g (201–270 V) | 35 mA/g | 35 mA/g |

Measurement conditions for capacitance and ESR (after washing and drying)

| | | | |
|-------------|---------------------------------|---------------------------------|---------------------------------|
| Electrolyte | 10 wt-% H_3PO_4 | 10 wt-% H_3PO_4 | 10 wt-% H_3PO_4 |
| Temperature | $23 \pm 2^\circ\text{C}$ | $23 \pm 2^\circ\text{C}$ | $23 \pm 2^\circ\text{C}$ |
| D.C. Bias | 2,0 V | 2,0 V | 2,0 V |
| A.C. Signal | 0,5 V | 0,5 V | 0,5 V |
| Frequency | 120 cps | 120 cps | 120 cps |

Measurement conditions for leakage current

| | | | |
|---------------|---------------------------------|---------------------------------|---------------------------------|
| Electrolyte | 10 wt-% H_3PO_4 | 10 wt-% H_3PO_4 | 10 wt-% H_3PO_4 |
| Temperature | $23 \pm 2^\circ\text{C}$ | $23 \pm 2^\circ\text{C}$ | $23 \pm 2^\circ\text{C}$ |
| Voltage | 240 V | 140 V | 70 V (47 V) |
| Charging time | 2 min | 2 min | 2 min |

Measurement conditions for dissipation factor (after washing and drying)

| | | | |
|-------------|---------------------------------|---------------------------------|---------------------------------|
| Electrolyte | 10 wt-% H_3PO_4 | 10 wt-% H_3PO_4 | 10 wt-% H_3PO_4 |
| Temperature | $23 \pm 2^\circ\text{C}$ | $23 \pm 2^\circ\text{C}$ | $23 \pm 2^\circ\text{C}$ |
| D.C. Bias | 2,0 V | 2,0 V | 2,0 V |
| A.C. Signal | 0,5 V | 0,5 V | 0,5 V |
| Frequency | 120 cps | 120 cps | 120 cps |

Measurement conditions for breakdown voltage

| | | | |
|-------------|--------------------------------|--------------------------------|--------------------------------|
| Electrolyte | 1 wt-% H_3PO_4 | 1 wt-% H_3PO_4 | 1 wt-% H_3PO_4 |
| Temperature | $90 \pm 2^\circ\text{C}$ | $90 \pm 2^\circ\text{C}$ | $90 \pm 2^\circ\text{C}$ |
| Current | 35 mA/g | 35 mA/g | 35 mA/g |

Other testing conditions can be applied according to customers requirements

**Tantalum Powder Capacitor Grade
Grade PL-8000**

PL-8000

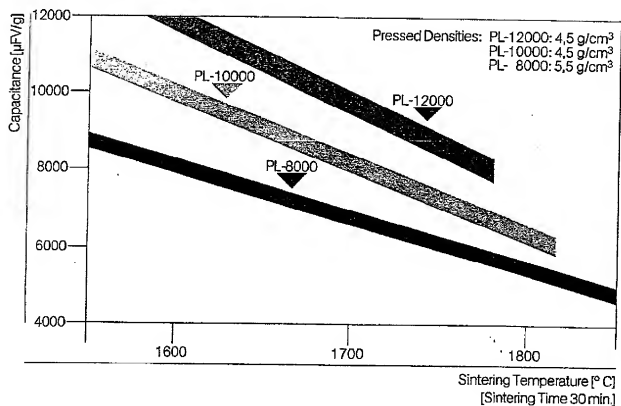
Metallurgical

PL-8000 represents the powder with the lowest yield of capacitance per gram and the highest working voltage in the PL-series. It has a good flowability and permits pressed densities down to 5.0 g/cm³. PL-8000 is recommended especially for the production of medium voltage 35 V capacitors.

Recommended conditions for processing

Sintering temperature: 1600 – 1850°C
Sintering time: 15 – 30 minutes
Pressed density: 5,0 – 6,0 g/cm³
can also be pressed
without binder
Yield of capacitance: 5000 – 8000 μ FV/g (Wet test)
Working voltage: up to 35 V

Typical Capacitance



Typical Electrical Characteristics

| Grade | μ FV/g | μ FV/ cm ³ | Sintering Temperature (°C) | VBD (V) | DCL (nA/ μ FV) |
|---------|------------|------------------------------|----------------------------------|------------|-----------------------|
| PL-8000 | 8000 | 47500 | 1600 | 180 | 0,4 |
| PL-8000 | 6800 | 44000 | 1700 | 200 | 0,30 |
| PL-8000 | 5500 | 38500 | 1800 | 220 | 0,2 |

Anode weight: 0,4 g, Pressed density: 5,5 g/cm³
Sintering time: 30 min, Formation voltage: 100 V

PL-8000

Typical Chemical Analysis

| Elements | H | N | O | C | Fe | Nb | Si | Ti | W | Mo |
|----------|----|----|------|----|----|----|----|----|----|----|
| ppm | 10 | 75 | 2000 | 75 | 50 | 50 | 50 | 10 | 20 | 20 |

Typical Physical Characteristics

Scott Density (according to ASTM No. B 329-76)

g/in³: 30 -40

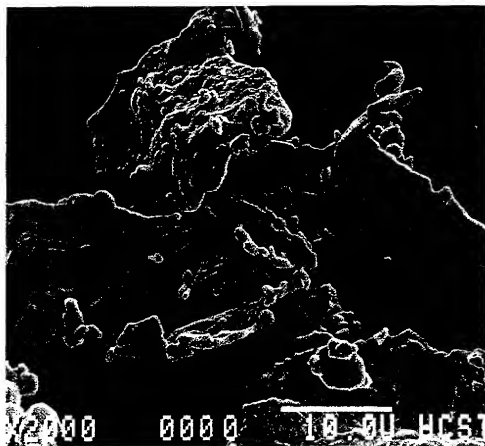
g/cm³: 1,8- 2,4

Fisher Sub-Sieve Size (according to ASTM No. B 330-82)

µm: 4,0 - 5,5

Screen Distribution (according to ASTM No. B 214-76, E 11)

| + 200 mesh | -200+325 mesh | -325 mesh |
|------------|---------------|-----------|
| 20-50% | 10-30% | 40-60% |



PL-8000

Scanning electron micrograph

Tantalum Powder Capacitor Grade Grade PL-10000

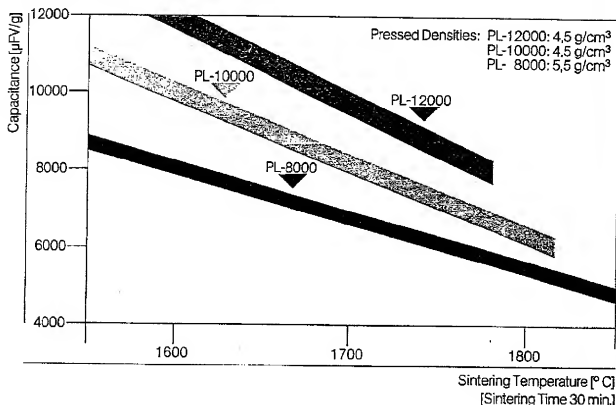
PL 10000

PL-10000 was the first powder with a yield of capacitance of more than 10000 $\mu\text{FV/g}$. Its excellent physical properties permit pressed densities down to 4.0 g/cm^3 . PL-10000 is recommended for the production of capacitors up to 35 V.

Recommended conditions for processing

Sintering temperature: 1600–1750°C
Sintering time: 15 –30 minutes
Pressed density: 4.0 – 5.0 g/cm^3
can also be pressed without binder
Yield of capacitance: 7500–10000 $\mu\text{FV/g}$ (Wet test)
Working voltage: up to 35 V

Typical Capacitance



Typical Electrical Characteristics

| Grade | $\mu\text{FV/g}$ | $\mu\text{FV/cm}^3$ | Sintering Temperature (°C) | VBD (V) | DCL (nA/ μFV) |
|----------|------------------|---------------------|----------------------------|---------|---------------------------|
| PL-10000 | 10000 | 49000 | 1600 | 170 | 0.5 |
| PL-10000 | 9100 | 47000 | 1650 | 180 | 0.4 |
| PL-10000 | 8200 | 45000 | 1700 | 190 | 0.3 |

Anode weight: 0.4 g, Pressed density: 4.5 g/cm^3
Sintering time: 30 min, Formation voltage: 100 V

PL-10000

Typical Chemical Analysis

| Elements | H | N | O | C | Fe | Nb | Si | Ti | W | Mo |
|----------|----|----|------|----|----|----|----|----|----|----|
| ppm | 10 | 75 | 2400 | 75 | 50 | 50 | 50 | 10 | 20 | 20 |

Typical Physical Characteristics

Scott Density (according to ASTM No. B 329-76)

g/in³ : 20 - 35

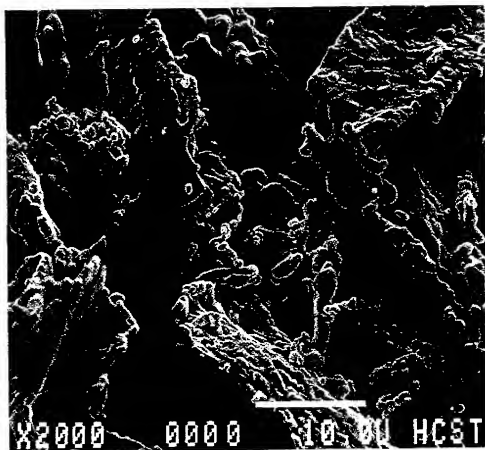
g/cm³ : 1,2 - 2,1

Fisher Sub-Sieve Size (according to ASTM No. B 330-82)

µm: 2,5 - 4,5

Screen Distribution (according to ASTM No. B 214-76, E 11)

| +200 mesh | -200+325 mesh | -325 mesh |
|-----------|---------------|-----------|
| 10-30% | 10-30% | 40-60% |



PL-10000

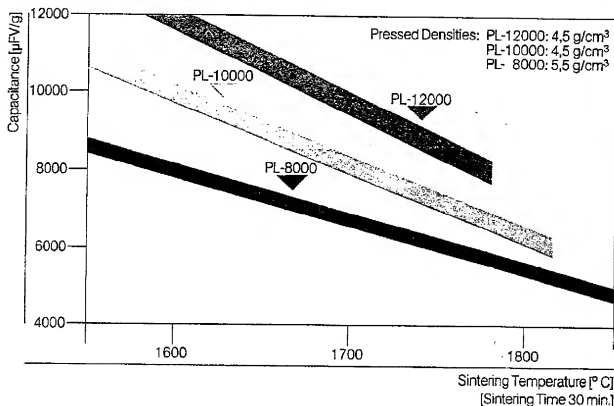
Scanning electron micrograph

PL-12000 today represents the powder with the highest yield of capacitance at normal sinter conditions of minimum 1600 °C, 30 min (12000 $\mu\text{FV/g}$). Its high green strength also permits pressed densities of 4,0 – 5,0 g/cm³. PL-12000 corresponds to the data of PL-10000 as to its processing qualities.

Recommended conditions for processing

Sintering temperature: 1600 – 1750°C
Sintering time: 15 – 30 minutes
Pressed density: 4,0 – 5,0 g/cm³
can be pressed without binder
Yield of capacitance: 9000 – 12000 $\mu\text{FV/g}$ (Wet test)
Working voltage up to 35 V

Typical Capacitance



Typical Electrical Characteristics

| Grade | $\mu\text{FV/g}$ | $\mu\text{FV/cm}^3$ | Sintering Temperature (°C) | VBD (V) | DCL (nA/ μFV) |
|----------|------------------|---------------------|----------------------------|---------|---------------------------|
| PL-12000 | 12000 | 58000 | 1600 | 160 | 0,5 |
| PL-12000 | 10700 | 53500 | 1650 | 170 | 0,35 |
| PL-12000 | 9800 | 50000 | 1700 | 180 | 0,25 |

Anode weight: 0,4 g, Pressed density: 4,5 g/cm³
Sintering time: 30 min, Formation voltage: 100 V

PL-12000

Typical Chemical Analysis

| Elements | H | N | O | C | Fe | Nb | Si | Ti | W | Mo |
|----------|----|-----|------|-----|----|----|----|----|----|----|
| ppm | 10 | 200 | 2700 | 100 | 50 | 50 | 50 | 10 | 20 | 20 |

Typical Physical Characteristics

Scott Density (according to ASTM: B 329-76)

g/in³ : 15 - 25

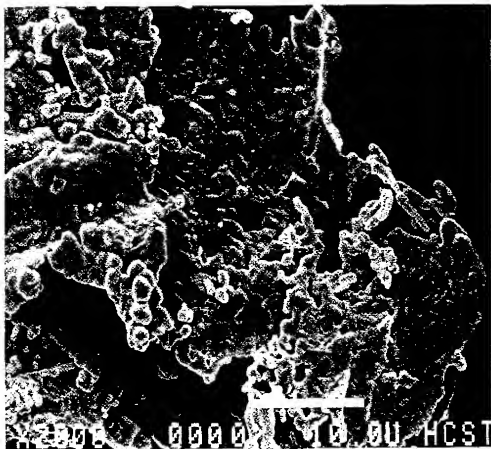
g/cm³: 0,9 - 1,5

Fisher Sub-Sieve Size (according to ASTM No. B 330-82)

µm: 2,0 - 3,5

Screen Distribution (according to ASTM: B 214-76, E 11)

| +200 mesh | -200+325 mesh | -325 mesh |
|-----------|---------------|-----------|
| 10-30% | 10-30% | 40-60% |



PL-12000

Scanning electron micrograph

**Tantalum Powder Capacitor Grade
Grade PL-18000 R**

PL-18000 R

PL-18000 R is a consistent improvement over PL-18000 with regard to better physical properties like flowability and green strength at low pressed density.

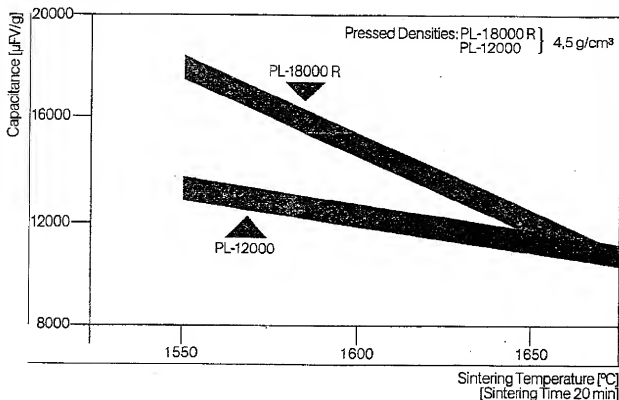
Further on it can be characterized by an outstanding low level of oxygen which has to be related to the high capacitance of this powder.

Because of the improved stability of PL-18000 R particles preparation of pellets is possible at a green density of 4,5 g/cm³.

Recommended conditions for processing

Sintering temperature: 1550–1700 °C
Sintering time: 5 –20 minutes
Pressed density: 4,5–5,5 g/cm³
can be pressed without binder
Yield of capacitance: 13000–18000 µFV/g
(wet test)
Working voltage: up to 25 V

Typical Capacitance



Typical Electrical Characteristics

| Grade | µFV/g | µFV/ cm³ | Sintering Temperature (°C) | VBD DCL (V) (nA/µFV) |
|------------------|-------|-------------|----------------------------------|-------------------------|
| PL-18000 R 18000 | 90000 | 1550 | 120 | 0,3 |
| PL-18000 R 15000 | 95000 | 1600 | 130 | 0,3 |
| PL-18000 R 12000 | 77500 | 1650 | 140 | 0,2 |

Anode weight: 0,2 g, Pressed density: 4,5 g/cm³
Sintering time: 20 min. Formation voltage: 70 V

PL-18000 R

Typical Chemical Analysis

| Elements | N | O | C | Fe | Nb | Si | Ti | W | Mo |
|----------|-----|------|----|----|----|----|----|----|----|
| ppm | 200 | 1700 | 90 | 60 | 50 | 50 | 10 | 20 | 20 |

Typical Physical Characteristics

Scott Density (according to ASTM: B 329-76)

g/in³ : 15-20

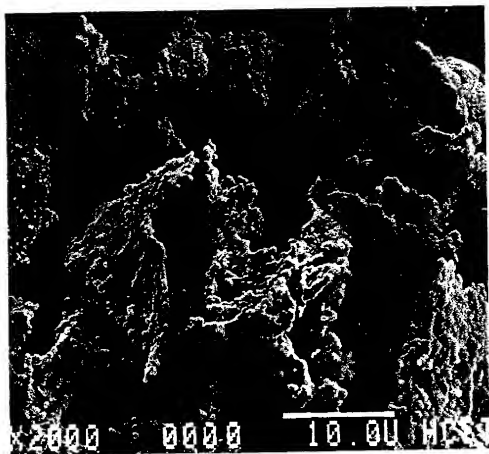
g/cm³ : 0,9-1,3

Fisher Sub-Sieve Size (according to ASTM: B 330-82)

µm : 0,9-1,8

Screen Distribution (according to ASTM: B 214-76, E11

100%-45 mesh



PL-18000 R

Scanning electron micrograph

**Tantalum Powder Capacitor Grade
Grade PL-22000**

PL-22000

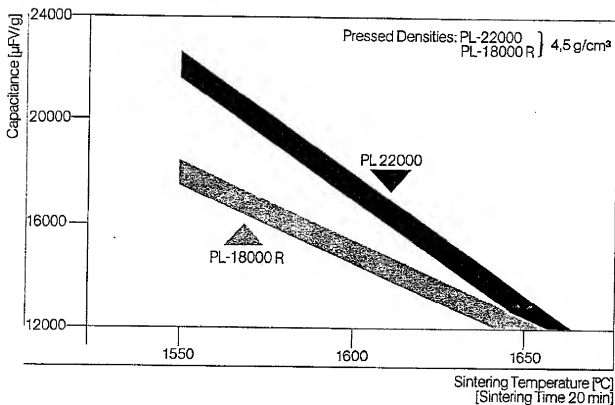
PL-22000 combines all the qualities of the PL-series with the highest yield of capacitance presently available in the market.

Its remarkably high yield of capacitance per unit volume should allow a further miniturization of capacitors under fixed conditions.

Recommended conditions for processing

Sintering temperature: 1500–1700 °C
Sintering time: 5–20 minutes
Pressed density: 4.5–5.5 g/cm³
can be pressed without binder
Yield of capacitance: 15000–22000 µFV/g
(wet test)
Working voltage: up to 25 V

Typical Capacitance



Typical Electrical Characteristics

| Grade | µFV/g | µFV/ cm ³ | Sintering Temperature (°C) | VBD DCL (V) (nA/µFV) |
|----------------|--------|-------------------------|----------------------------------|-------------------------|
| PL-22000 22000 | 110000 | 1550 | 120 | 0,3 |
| PL-22000 17000 | 95000 | 1600 | 130 | 0,3 |
| PL-22000 13000 | 77500 | 1650 | 140 | 0,2 |

Anode weight: 0,2 g, Pressed density: 4,5 g/cm³
Sintering time: 20 min. Formation voltage: 70 V

PL-22000

Typical Chemical Analysis

| Elements | N | O | C | Fe | Nb | Si | Ti | W | Mo |
|----------|-----|------|-----|----|----|----|----|----|----|
| ppm | 200 | 2000 | 100 | 60 | 50 | 50 | 10 | 20 | 20 |

Typical Physical Characteristics

Scott Density (according to ASTM: B 329-76)

g/in³ : 15-20

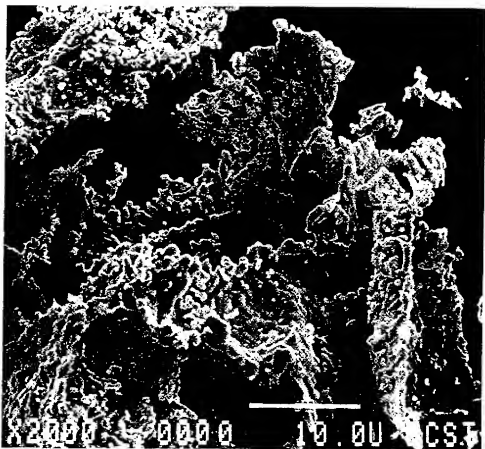
g/cm³: 0,9-1,3

Fisher Sub-Sieve Size (according to ASTM: B 330-82)

μm : 0,9-1,3

Screen Distribution (according to ASTM: B 214-76, E 11

100%-45 mesh



PL-22000

Scanning electron micrograph

Tantalum Powder Capacitor Grade Grade 660

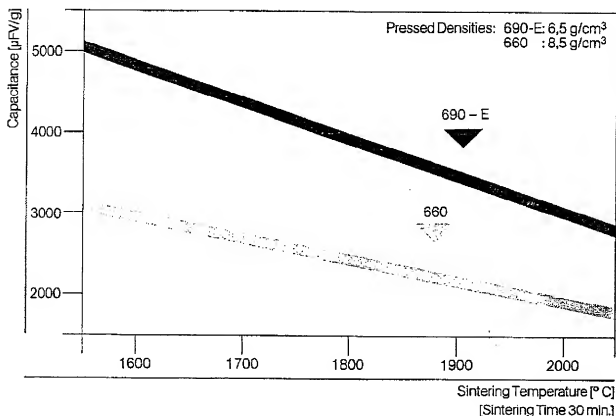
The application of grade 660 is in the high to highest working voltage area.

Because of the purity (EB-melting), this powder is very important for the production of capacitors with high reliability (MIL-Spec. No. C 39003)

Recommended conditions for processing

Sintering temperature: 1850–2000°C
Sintering time: 30–40 minutes
Pressed density: 7,5–9,5 g/cm³
Yield of capacitance: 1900–2400 μ FV/g
Working voltage: 50 V

Typical Capacitance



Typical Electrical Characteristics

| Grade | μ FV/g | μ FV/ cm ³ | Sintering Temperature (°C) | VBD (V) | DCL (nA/ μ FV) |
|-------|------------|------------------------------|----------------------------------|------------|-----------------------|
| 660 | 2350 | 21800 | 1850 | 230 | 0,50 |
| 660 | 2000 | 19500 | 2000 | 250 | 0,30 |

Anode weight: 4,0 g, Pressed density: 8,5 g/cm³
Sintering time: 30 min, Formation voltage: 200 V

Typical Chemical Analysis

| Elements | H | N | O | C | Fe | Nb | Si | Ti | W | Mo |
|----------|----|----|------|----|----|----|----|----|----|----|
| ppm | 10 | 35 | 1300 | 30 | 20 | 35 | 10 | 5 | 10 | 10 |

Typical Physical Characteristics

Scott Density (according to ASTM No. B 329-76)

g/in³ : 85 -100

g/cm³: 5,2- 6,1

Fisher Sub-Sieve Size (according to ASTM No. B 330-82)

µm : 8,0- 10,0

Screen Distribution (according to ASTM No. B 214-76, E11)

| | | |
|-----------|---------------|-----------|
| +200 mesh | -200+325 mesh | -325 mesh |
|-----------|---------------|-----------|

| | | |
|--------|--------|--------|
| 10-20% | 10-20% | 50-80% |
|--------|--------|--------|

Tantalum Powder Capacitor Grade Grade 690-E

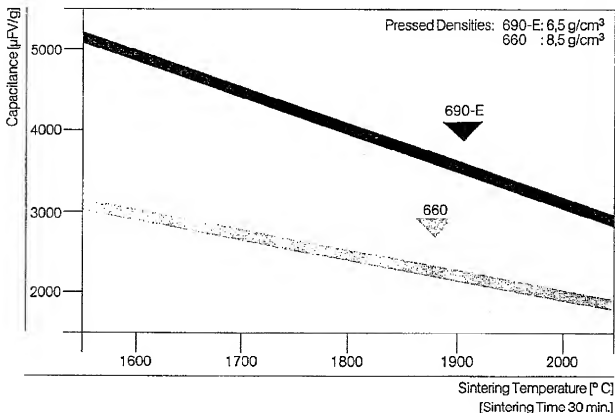
690-E

690-E is an improved EB-melted high capacitive powder. Its outstanding physical properties are: formability without binder, very good green strength and good flowability.

Recommended conditions for processing

Sintering temperature: 1600–1950°C
Sintering time: 15–40 minutes,
can be pressed without binder
Pressed density: 6.5–7.5 g/cm³
Yield of capacitance: 3000–5000 μFV/g
Working voltage: up to 50 V

Typical Capacitance



Typical Electrical Characteristics

| Grade | μFV/g | μFV/ cm ³ | Sintering Temperature (°C) | VBD (V) | DCL (nA/μFV) |
|-------|-------|-------------------------|----------------------------------|------------|-----------------|
| 690-E | 4650 | 33400 | 1650 | 190 | 0.4 |
| 690-E | 3750 | 32000 | 1650 | 240 | 0.3 |

Anode weight: 1.0 g, Pressed density: 7.0 g/cm³
Sintering time: 30 min, Formation voltage: 200 V

690-E

Typical Chemical Analysis

| Elements | H | N | O | C | Fe | Nb | Si | Ti | W | Mo |
|----------|----|----|------|----|----|----|----|----|----|----|
| ppm | 10 | 35 | 1600 | 40 | 25 | 35 | 10 | 5 | 10 | 10 |

Typical Physical Characteristics

Scott Density (according to ASTM: B 329-76)

g/in³ : 60 - 75

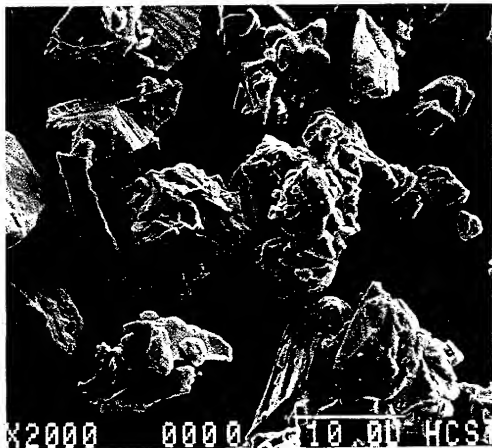
g/cm³ : 3,6 - 4,6

Fisher Sub-Sieve Size (according to ASTM: B 330-82)

µm : 7-12

Screen Distribution (according to ASTM: B 214-76, E 11)

| +200 mesh | -200+325 mesh | -325 mesh |
|-----------|---------------|-----------|
| 40-85% | 5-15% | 30-50% |

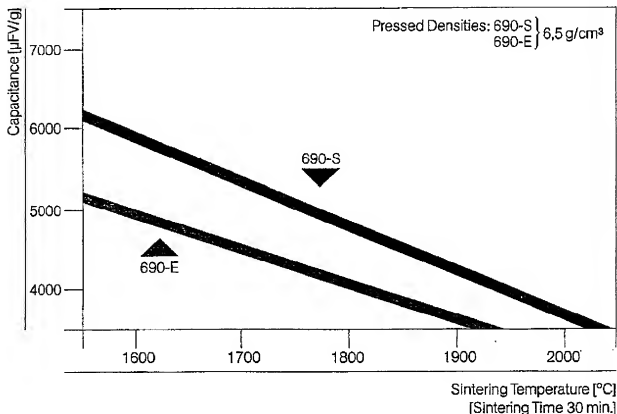


690-S is a newly developed EB-melted high capacitance powder with all the physical properties of 690-E, such as excellent pressing characteristics without binder, good green strength and good flowability.

Recommended conditions for processing

Sintering temperature: 1600–1850°C
Sintering time: 30–40 minutes
Pressed density: 6,5–7,5 g/cm³
can be pressed without binder
Yield of capacitance: 4500–6000 μ FV/g
Working voltage: 35 V and over

Typical Capacitance



Typical Electrical Characteristics

| Grade | μ FV/g | μ FV/ cm ³ | Sintering Temperature (°C) | VBD (V) | DCL (nA/ μ FV) |
|-------|------------|------------------------------|----------------------------------|------------|-----------------------|
| 690-S | 5650 | 49000 | 1650 | 190 | 0,4 |
| 690-S | 4250 | 41000 | 1850 | 240 | 0,3 |

Anode weight: 1,0 g, Pressed density: 7,0 g/cm³
Sintering time: 30 min, Formation voltage: 200 V

690-S

Typical Chemical Analysis

| Elements | H | N | O | C | Fe | Nb | Si | Ti | W | Mo |
|----------|----|----|------|----|----|----|----|----|----|----|
| ppm | 10 | 35 | 1700 | 40 | 25 | 35 | 10 | 5 | 10 | 10 |

Typical Physical Characteristics

Scott Density (according to ASTM: B 329-76)

g/in³ : 60 -75

g/cm³: 3,6-4,6

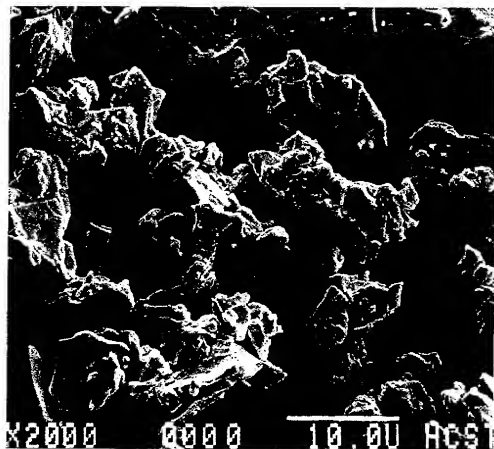
Fisher Sub-Sieve Size (according to ASTM: B 330-82)

μm : 4-9

Screen Distribution (according to ASTM: B 214-76, E 11)

| | | |
|-----------|---------------|-----------|
| +200 mesh | -200-325 mesh | -325 mesh |
|-----------|---------------|-----------|

| | | |
|--------|-------|--------|
| 30-55% | 3-10% | 40-60% |
|--------|-------|--------|



**Tantalum Powder Capacitor Grade
Grade 900-HC**

900-HC

Metallurgical

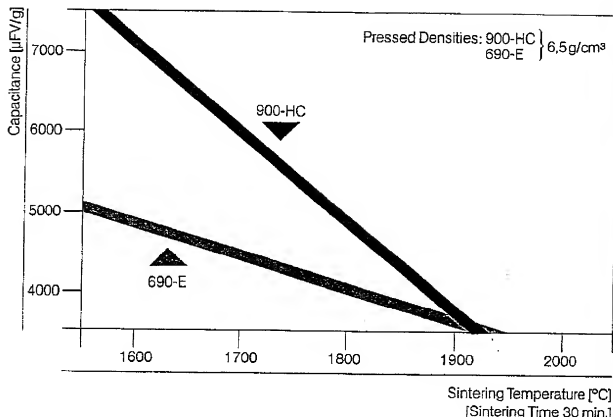
900-HC represents a new class of EB-melted high capacitance powders. The capacitance increase compared to grade 690-E is about 25% at 1700°C sintering temperature.

The application of 900-HC is in the high working voltage area.

Recommended conditions for processing

Sintering temperature: 1600–1850°C
Sintering time: 20–40 minutes
Pressed density: 6,5–7,5 g/cm³
can be pressed without binder
Yield of capacitance: 4500–7000 µFV/g
Working voltage: 35 V and over

Typical Capacitance



Typical Electrical Characteristics

| Grade | µFV/g | µFV/ cm ³ | Sintering Temperature (°C) | VBD (V) | DCL (nA/µFV) |
|--------|-------|-------------------------|----------------------------------|------------|-----------------|
| 900-HC | 6100 | 50000 | 1700 | 210 | 0,4 |
| 900-HC | 4900 | 44000 | 1800 | 225 | 0,3 |

Anode weight: 1,0 g, Pressed density: 6,5 g/cm³
Sintering time: 30 min, Formation voltage: 200 V

900-HC

Typical Chemical Analysis

| Elements | H | N | O | C | Fe | Nb | Si | Ti | W | Mo |
|----------|----|----|------|----|----|----|----|----|----|----|
| ppm | 10 | 50 | 2000 | 40 | 35 | 35 | 10 | 5 | 10 | 10 |

Typical Physical Characteristics

Scott Density (according to ASTM: B 329-76)

g/in³ : 55 -70

g/cm³ : 3,3-4,3

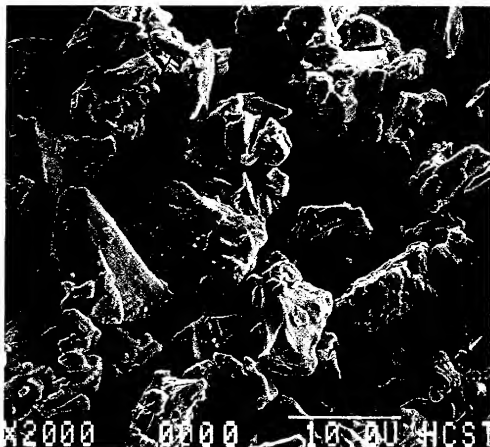
Fisher Sub-Sieve Size (according to ASTM: B 330-82)

μm : 4-7

Screen Distribution (according to ASTM: B 214-76, E 11)

-40 mesh -325 mesh

100% 30-50%



Tantalum Powder Capacitor Grade Special Grade

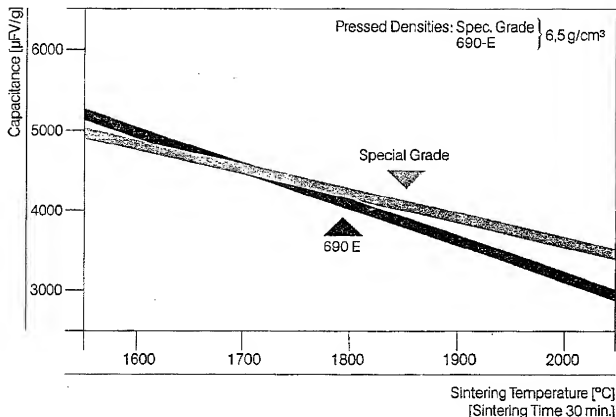
Special Grade is an EB-melted high capacitance powder with smaller temperature-capacitance-coefficient compared to grade 690-E.

The outstanding physical properties such as good flowability and high pellet green strength are almost identical to grade 690-E.

Recommended conditions for processing

Sintering temperature: 1600–1950°C
Sintering time: 15–40 minutes
Pressed density: 6.5–7.5 g/cm³
can be pressed without binder
Yield of capacitance: 3000–5000 $\mu\text{FV/g}$
Working voltage: 35 V and over

Typical Capacitance



Typical Electrical Characteristics

| Grade | $\mu\text{FV/g}$ | $\mu\text{FV/cm}^3$ | Sintering Temperature ($^{\circ}\text{C}$) | VBD (V) | DCL (nA/ μFV) |
|------------------|------------------|---------------------|--|---------|---------------------------|
| Spec. Grade 4600 | 33000 | 1650 | 195 | 0.4 | |
| Spec. Grade 3850 | 32500 | 1850 | 245 | 0.3 | |

Anode weight: 1.0 g, Pressed density: 7.0 g/cm³
Sintering time: 30 min. Formation voltage: 200 V

Special
Grade

Special Grade

Typical Chemical Analysis

| Elements | H | N | O | C | Fe | Nb | Si | Ti | W | Mo |
|----------|----|----|------|----|----|----|----|----|----|----|
| ppm | 10 | 35 | 1600 | 30 | 25 | 35 | 10 | 5 | 10 | 10 |

Typical Physical Characteristics

Scott Density (according to ASTM: B 329-76)

g/in³ : 60 -70

g/cm³ : 3,6-4,3

Fisher Sub-Sieve Size (according to ASTM: B 330-82)

μm : 7-12

Screen Distribution (according to ASTM: B 214-76, E 11)

-40 mesh -325 mesh

100% 30-50%



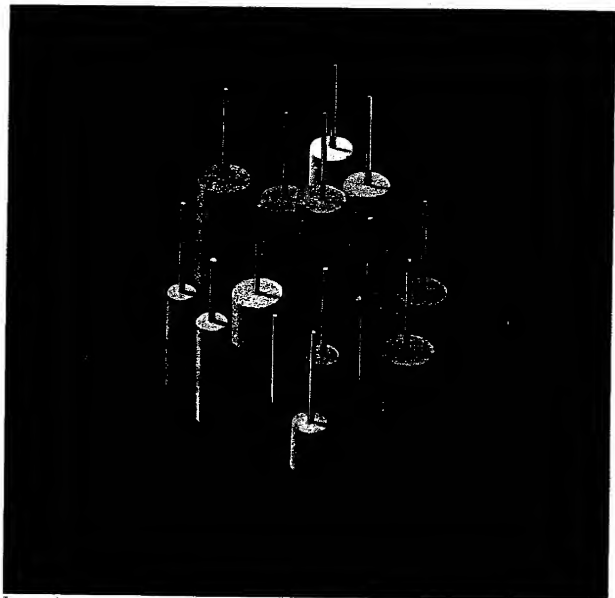
Sintered Tantalum Anodes

Hermann C. Starck Berlin have been supplying sintered anodes for wet and dry capacitors since 1969.

Years of experience in producing capacitor grade tantalum powder also enables us to produce high quality sintered anodes. The aim of our anode production is to support existing production of our customers and not to compete with our customers.

In view of the enormous variety of types, data sheets for the individual anode types are not available. We are generally prepared to meet anode specifications within the following data.

| | |
|--------------------|---|
| Anode form: | cylindrical, rectangular |
| Anode diameter: | 1,0 – 10,0 mm |
| Anode length: | 0,5 – 20 mm |
| Wire diameter: | 0,25 – 0,8 mm |
| Wire length: | 10 – 22 mm |
| | (Standard figures: 10 mm, 15 mm, 20 mm) |
| Wire lead: | central, asymmetric |
| Capacitance: | 0,006 – 680 μ F |
| Formation voltage: | up to 270 V |



Tantalum Metal Metallurgical Grade Powder

Scope: This specification covers the requirement for tantalum powder used for powder metallurgy.

Physical Analysis

Grain size: 100%-70 mesh
Scott Density: 60-100 g/inch³
(according to ASTM: B 329-76)
Fisher Sub-Sieve Size: 6-16 μ m
(according to ASTM: B 330-76)

Chemical Analysis (max values in ppm):

| H | C | N | O | AL | Si |
|-----|-----|-----|------|----|-----|
| 100 | 200 | 200 | 2000 | 50 | 200 |
| Cb | W | Mo | Fe | Cr | Ni |
| 200 | 200 | 200 | 200 | 50 | 50 |

Powder out of this grade can be used for tantalum mill products.

Especially in case of wire used in leads for tantalum capacitors and component parts, properties like resistance to grain growth during high temperature sintering and low leakage current are required.

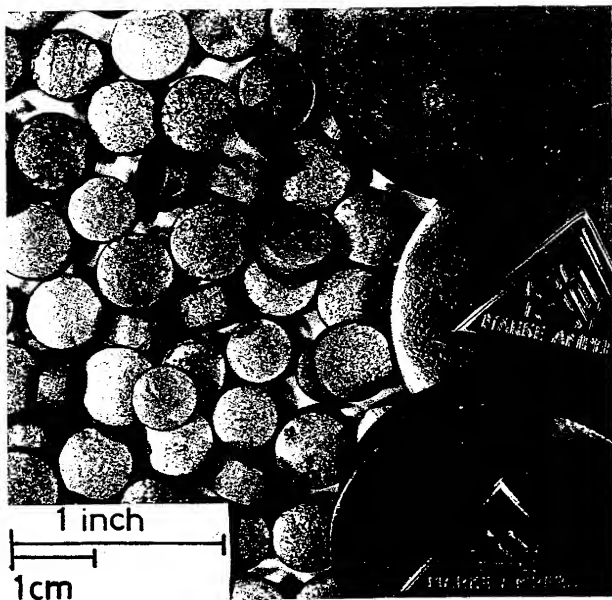
There are two starting materials commonly used for mill products: electron beam-arc-melted ingots or pressed and sintered powder bars.

In case powder is used the powder has to bear all the characteristics of the final product.

Metallurgical grade powder by **HCST** is recommended for this purpose.

Regarding the specification we are prepared to meet customers' requirements.

Metallurgical Grade Powder



Tantalum Metal Metallurgical Grade Compacts

Scope: This specification covers the requirement for tantalum to produce high-quality alloys by vacuum melting.

I. High Purity Grade — min. 99,8% Ta

| H | C | N | O | Al | Si | S | Ti | |
|-----|-----|-----|-----|----|-----|----|----|----|
| 10 | 300 | 150 | 600 | 50 | 100 | 20 | 30 | |
| Fe | Se | Cb | Ag | Sn | Sb | Te | Pb | Br |
| 150 | 10 | 200 | 10 | 10 | 5 | 10 | 10 | 5 |

II. Special Grade — min. 99,7% Ta

| H | C | N | O | Al | Si | S | Ti | |
|-----|-----|-----|------|----|-----|----|----|----|
| 10 | 500 | 300 | 1500 | 50 | 200 | 20 | 30 | |
| Fe | Se | Cb | Ag | Sn | Sb | Te | Pb | Bi |
| 150 | 10 | 200 | 10 | 10 | 5 | 10 | 10 | 5 |

Size

High purity grade (99,8% Ta) and
Special grade (99,7% Ta) are available in tablets

Dimensions for Tablets

Diameter 5–10 mm (0,2"–0,4")
Length 5–10 mm (0,2"–0,4")

Diameter 40 mm (1,57")
Length 10–15 mm (0,4"–0,6")

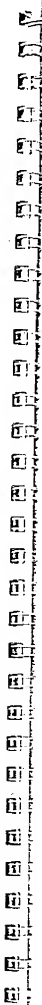
Packaging

The standard package is
100 lbs per drum

Metallurgical Grade Compacts

The technical information of this data sheet represents our present knowledge. It does not form part of any sales contracts as guaranteed properties of the delivered material. Our delivery and sales conditions apply to all

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12) Related Proceedings Appendix

None.